TEXTILE RESEARCH

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TEXTILE RESEARCH

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In This Month's Issue

Studies in Water-Repellent Pyridinium Compounds

Factors affecting the efficiency of that class of chemical compounds known as synthetic permanent water-repellents are discussed. For example, it is shown that slight differences in chemical structure have considerable influence on the water repellency. The production of this type of finish is quite complicated, requiring skilled labor, special and exactly controllable equipment, and well defined raw materials to obtain good results. (See page 242.)

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N. C. State College Textile School

A new perspective on the relationship of the textile school to industry is reflected in the expanded programs of some of our textile schools. (See page 246.)

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Economics and Technique

Douglas Woolf explains the importance of economics in the field of textile research. (See page 252.)

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Statistical Program

A statistical program is developing which is intended to provide both Government and business with the factual information on production and distribution needed not only during the difficult period of transition from war to civilian production, but also for efficient continuing operation. (See page 257.)

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The Story of Coated Fabrics

The development of methods for applications of rubberized coatings and the newer synthetic resin coatings is described. (See page 260.)

Studies in Water-Repellent Pyridinium Compounds

By ERNEST ZERNER*
and PETER I. POLLAK

The textile industry knows numerous types of chemicals which, when applied to cloth in a suitable manner, will render the fabric water repellent. The oldest type of waterproofing compounds found little application, since they completely altered the hand and feel of the material and sealed the pores of the fabric.

In the last 20 years the development of water-repellent chemicals has led to two types in use now in the finishing industry. One type consists of wax-aluminum acetate (or formate) emulsions of all kinds which, applied to the material in the right way, will render it water repellent without sealing the pores. This type of chemical is manufactured at comparatively small cost and its application by the finisher is simple and cheap. No complicated equipment is required to produce adequate results.

The chief disadvantage of this type of repellent lies in its performance. The finish consists essentially of a thin layer of hydrophobic material combined with the fabric in a physical way only. It is therefore more or less completely removed by laundering or dry cleaning. Materials finished with this wax-aluminum acetate-emulsion type show good water-repellency but have to be refinished after each cleansing process. On the other hand, the physical nature of the finish makes it possible to apply this finish to all textiles regardless of their chemical nature, its effectiveness being only limited by the size of the pores of the material to be finished.

In contrast to this class of water-repellents, there exists a large body of chemical compounds known as synthetic permanent water-repellents. This type of finish, which has its cradle in various research laboratories of this and other countries, is most satisfactory in performance. Although there exist a great number of patents in this specific field of organic synthetic research—a fact which confuses the novice—it is apparent that certain characteristics in the chemical nature of the compounds are quite universal and are generally accepted as yielding the best results. However, it appears as explained later that sometimes slight differences in chemical structure have a considerable influence on the water-repellency.

The production of this type of finish is comparatively complicated, requires skilled labor, expensive equipment, and well defined raw materials. In general, a modern concept of chemical manufacturing is necessary to assure successful production. The finisher needs special and exactly controllable equipment to obtain good results.

* Ernest Zerner, director of research, and Peter I. Pollak, research chemist, Warwick Chemical Corporation, Research Laboratories, New York, N. Y.

The synthetic permanent water-repellents are defined as chemical compounds. Their molecule contains a hydrophobic part which serves as the water-repellent, and consists, in most cases, of fatty acid derivatives. This hydrophobic part of the molecule is in some way combined with a polar group of varied chemical nature. This polar part has to react with the hydroxyl groups of the cellulose and thus to combine chemically the hydrophobic repellent with the cellulose fiber of the fabric, forming a continuous monomolecular water-repellent layer. The necessary reaction is effected by curing at a temperature around 300° F for a few minutes. Besides water-repellency, the fabric acquires a very nice, soft feel with this treatment.

Due to the chemical nature of the bond between cellulose and water-repellent, the repellency is durable and not removed to any appreciable extent by laundering or dry cleaning.

In case of a fabric, where most of the hydroxyl groups of the cellulose are substituted, as in the case of cellulose acetate, none, or very little reaction, will occur between fiber and durable water-repellent; in other words, an otherwise excellent water-repellent of this type will not yield satisfactory results with acetate.

It is, of course, an advantage to use the water-repellent in an aqueous solution, or dispersion, which has to be stable at least for some hours. Therefore, the polar group is preferably built up in such a way that the whole compound is easily dispersible in water.

The permanent water-repellent compounds used at present in the textile industry are quaternary ammonium compounds of pyridine which are dispersible in water. At elevated temperatures, pyridine is split off and the rest of the molecule reacts with the hydroxyl groups of the cellulose to form ether-like compounds.

Taking stearamidomethylpyridiniumchloride as representative of these products we may formulate the reaction as follows:

$$C_{17}H_{35}CONHCH_2NC_5H_5Cl + \text{cellulose—OH} \rightarrow C_{17}H_{35}CONHCH_2\text{—O—cellulose} + C_5H_5NHCl$$

The quaternary ammonium hydroxide of the pyridinium compound may possibly be formed as an intermediate.

As previously mentioned it is essential that the quaternary ammonium compounds consist of a hydrophobic part (mostly a derivative of steric acid since lower molecular weight fatty acids such as lauric acid or unsaturated fatty acids are less satisfactory) and a polar group which is capable of forming a bond with the cellulose.

This polar group must be very reactive, a property which is present in the chloromethyl group, but not in the chloroethyl group. This is demonstrated by the repellency produced by the following compounds:

$C_{17}H_{35}CONHCH_2—NC_5H_5—Cl$ excellent repellency

$C_{17}H_{35}CONHCH_2CH_2—NC_5H_5—Cl$ [1] bad repellency

$$C_{17}H_{35}CONHCH(CH_3)—NC_5H_5—Cl$$ [2] medium repellency

We have synthesized numerous quaternary ammonium compounds and

[1] U.S.P.: 2,242,211 (1941). E. Haack to Heyden.

[2] Made by condensing stearamide with paraldehyde instead of paraformaldehyde.

found that small differences in internal molecular structure have great influence upon the water-repellent properties of the compounds. Striking is, for example, the difference in water-repellency between the following two compounds:

$C_{17}H_{35}CONCH_2—NC_5H_5—Cl$ [3]
CH_3 good repellency

$CH_3CONCH_2—NC_5H_5—Cl$
$C_{18}H_{37}$ bad repellency

The difference between the two compounds is that in the first compound the long chain fatty radical is connected with the carbonyl group, while in the second compound the hydrophobic part is connected with the nitrogen atom.

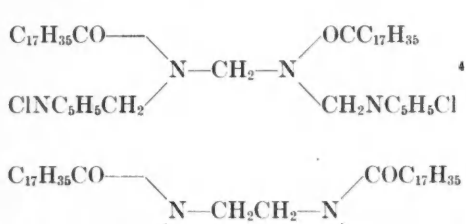
Another series of experiments showed the following interesting results:

ylene diamine [5] by chloromethylation with two moles of paraformaldehyde and gaseous hydrogen chloride.

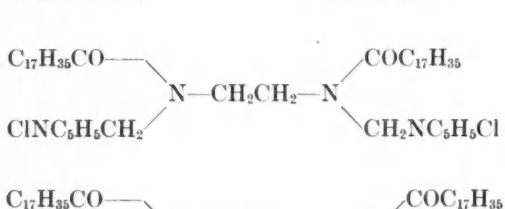
The ethylene homologue was made by condensing ethylene diamine with stearic acid to form the N,N'-distearoyl ethylene diamine, m.p. (alcohol): 139.5–140° C, which was subsequently chloromethylated.

Since the 1,3-diaminopropane was commercially not available it was prepared by the Gabriel method from 1,3-dibromopropane and potassium phthalimide.[6] It was subsequently condensed with stearic acid to N, N'-distearoyl trimethylene diamine, m.p. (Dioxane): 126–127° C, which was then chloromethylated.

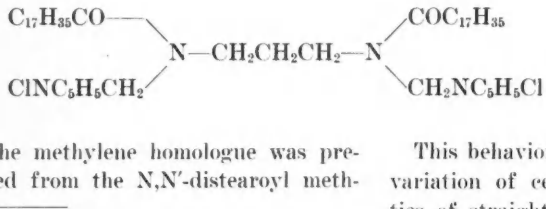
The results shown above indicate that unsatisfactory repellencies are obtained when the chain connecting the two amide radicals contains an even number of methylene groups.



[4] excellent repellency



bad repellency



good repellency

The methylene homologue was prepared from the N,N'-distearoyl meth-

This behavior has an analogy in the variation of certain physical properties of straight chain aliphatic homo-

[3] U.S.P.: 2,131,362 (1938). Baldwin and Piggott to Imperial Chemicals.

[4] U.S.P.: 2,306,185 (1942). J. Pike to Du Pont; U.S.P.: 2,213,741 (1943). M. Englemann and J. Pike to Du Pont.

[5] French Patent 782,330.

[6] Journal American Chemical Society, 1926, p. 2348.

logues and their derivatives. When plotting the melting points of the terminal dibromides of alkanes against the number of carbon atoms in the molecule we obtain a curve comparable to that obtained from the melting points of dibasic fatty acids as a function of the number of carbon atoms per molecule. (See Fig. 1.)

The cause of these variations is found in the steric arrangements of the chains of the molecules. From X-ray data and dipole moments it may be inferred that the dibasic fatty acids have the two carboxyl groups pointed in one direction if the molecule contains an odd number of carbon atoms, while the carboxyl groups point in opposite directions in a molecule containing an even number of carbon atoms. By analogy we may deduct the same picture for other terminal bifunctional fatty acid derivatives, and it may well be that bond formation, and therefore the repellency, is favored in the case where the two reactive polar groups point in one direction, in other words, where the number of methylene groups in the bridge between the two amide groups is odd.

TABLE I. Melting points and number of methylene groups per molecule.

Dibromides:	M.P. °C	No. of methylene groups per molecule
Methylene	— 53	1
Ethylene	+ 10	2
Trimethylene	— 34	3
Tetramethylene	+ 24	4
Pentamethylene	— 35	5
Dibasic Fatty Acids:	**M.P. °C**	**No. of methylene groups per molecule**
Malonic	136	1
Succinic	185	2
Glutaric	96	3
Adipic	151	4
Pimelic	103	5

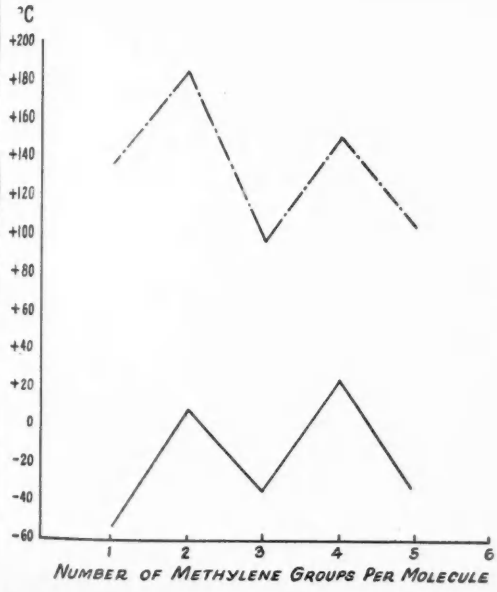


Fig. 1. Plot of melting points against number of methylene groups per molecule.
—.—.—: Dibasic acids
————: Dibromoalkanes

Emphasis is Placed Upon Research in Projected Program for

N. C. State College Textile School

By MALCOLM E. CAMPBELL*

A new perspective on the relationship of the textile school to industry is reflected in the expanded programs of some of our textile schools. In TEXTILE RESEARCH *for February the objectives of the Philadelphia Textile Institute were outlined. The following article from a recent address by Dean Campbell before the Piedmont Section, American Association of Textile Chemists and Colorists, describes the program at North Carolina State College. We hope to supply in these columns, information on the plans of other schools as they are developed.*

* * *

Training Men for Research

At the present time and in the past, the problem of obtaining personnel with adequate qualifications for textile research has been the most serious one in any program. With few exceptions the laboratories themselves have had to be the training schools for research workers. Young men and women well-grounded in chemistry, physics, or textile manufacturing must now spend long, unproductive periods in the work before they can become valuable assets to a research unit. I do not mean to say that this practical experience can be substantially replaced by any training that is now or will be available in the colleges and universities for many years to come. But with a teaching staff possessing not only an appreciation of research but also a background of experience in it, a textile school should be able to impart some invaluable training to the student who plans to take up research as his life's work.

Each textile school differs from the others in its objectives and its make-up as regards teaching staff and equipment. Upon those factors will depend the success with which these institutions can turn out students who are fitted to become productive in textile research after a minimum of experience following graduation.

I have referred to the possibilities of training young men and women as professional research workers. No doubt only a small proportion of the students studying textiles will possess the qualifications or even the desire to get into this field. It is difficult to imagine a person in any branch of the industry, however, to whom even a speaking acquaintance with textile research will not prove to be a valuable asset. The fruits of research will be a useless product indeed unless the men in production have an appreciation of its value, and an intense desire to apply it and

* Malcolm E. Campbell, dean, Textile School, North Carolina State College; formerly research manager, Textile Research Institute, Inc.

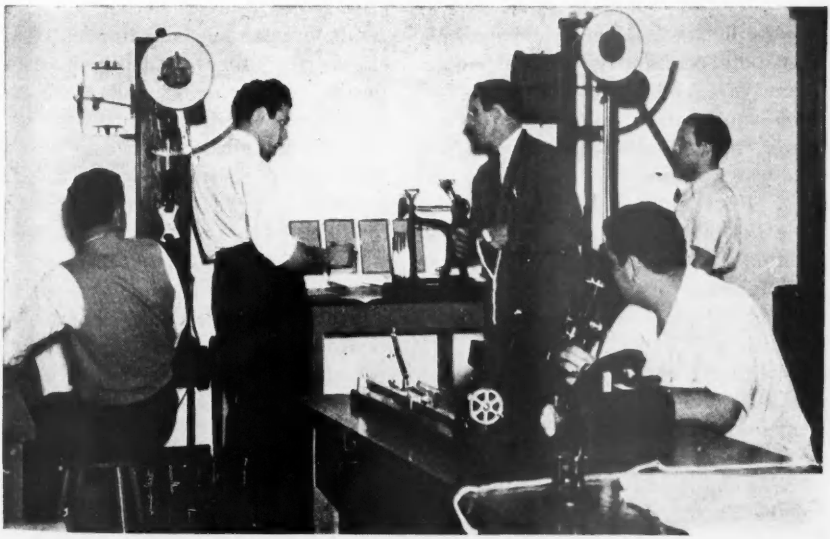
profit thereby in their own organizations. Therefore, to the greatest possible extent, a school should endeavor to acquaint all of its students with the possibilities and limitations of the tools of textile research; with the most modern methods of analyzing and interpreting the results of research; and lastly, with the identity, scope and objectives of the different organizations conducting textile research.

Staff and Facilities

In order to provide a basis for understanding the caliber of research instruction that our school may be able to impart (as well as the kind of research that we shall be able to pursue, which I shall discuss a little later) I shall describe briefly the kind of men we now have on our staff, and mention at least some of the equipment available at the school.

The head of our department of textile chemistry and dyeing is a man who has had 30 years' experience in teaching his subject, and, I believe, enjoys a reputation second to none in his field as a fine teacher and an excellent research man. He thinks in terms of today and tomorrow, instead of in the past, as is so frequently the case with men of such long experience. As head of our yarn manufacturing department we are fortunate in having a man who has had a fine engineering education, followed by many years of responsible experience both as a mill executive and as a research and developmental engineer. One of the key men in our weaving and designing department has had a considerable amount of experience in applied research in warp sizing and other phases of his field. The newly-appointed head of the knitting division is a trained mechanical engineer who has conducted research in European plants, and who has a number of mechanical developments in knitting to his credit. A fifth mem-

Fig. 1. Class in textile testing at N. C. State College Textile School.



ber of our staff, whose duties involve cotton manufacturing research as well as the rendering of technical assistance to mills, has worked previously on a number of textile projects. Other members of the textile faculty have had some experience in research, as well as in the production phases of the technical subjects they are teaching.

As for facilities and equipment, the following partial list will serve merely to indicate the scope of research possible at the school: In the textile chemistry and dyeing department, besides large- and small-scale bleaching and dyeing equipment, there is an unusually fine collection of microscopes, photomicrographic equipment, fiber cross-sectioning devices, a Fade-O-meter, a Launder-O-meter, pH equipment, drying ovens, and other specialized research apparatus. For physical testing and research, there is now being installed a precision air-conditioning system in our laboratories. Recent acquisitions in apparatus include a Suter-Webb fiber sorter, a Fibrograph, a Pressley fiber strength tester, several torsion balances, a power-driven yarn reel, both pendulum-type and inclined-plane yarn and fabric testers, a Moscrop automatic single-strand yarn tester, fabric abrasion testers, and air and water permeability testers for fabrics. Pilot laboratories are also available containing adequate machinery for spinning, weaving, knitting, and dyeing and finishing cotton, wool, and the various synthetic fibers under controlled conditions.

We therefore believe that the Textile School at State College is in an excellent position to give sound training in textile research methods to its students.

What has just been said with regard to the Textile School at State College is also true in greater or less degree with other textile schools in the country. The direction and pitch of their research training will depend upon the objectives of each institution, upon the quality of its teaching staff, and upon its facilities. As the demand for trained graduates in the field increases, and this is sure to occur, the schools will of necessity readjust their sights to this important requirement.

Conducting Research

So much for the training of students in research in the textile schools. And now the question: Should textile schools attempt seriously to carry on research programs themselves, and if so, of what general types? Again the answer will depend upon the peculiar set of circumstances existing in the individual institution, and in this connection, I shall confine my remarks to the Textile School at State College. Here, the answer is definitely yes. I say this in spite of the feeling that exists in some quarters that too many organizations already occupy the field, and that as a result, overlapping and duplication of effort are tending to stifle the possibilities that anything of real worth can result from additional effort. To anyone who has made a serious study of the question, it is obvious that there is no such thing as too much research, provided, of course, that those responsible for the various programs have the proper perspective and guard against a general muddle of the kind mentioned. Fortunately, such a possibility has already been effectively precluded by the organization of the Inter-Society Council for Textile Research. If the members of this Council each keep one foot on the ground, there need be no confusion; in fact, the various research

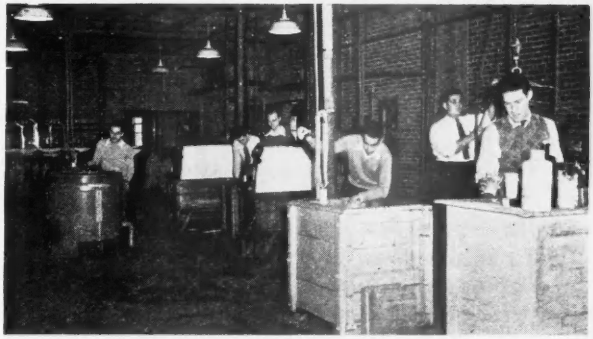


Fig. 2. Students working in dye laboratory, N. C. State College Textile School.

organizations can and, I believe, will cooperate and prove mutually helpful.

We shall conduct research primarily because we feel that we are equipped, mentally and physically, to contribute something useful to the industry through it, and I do not think this motive needs further expansion. A secondary motive, but one of importance to us, is that research keeps a teaching staff on its toes—protects them from the inevitable rut into which so many good teachers fall if they do not have something new to help maintain a proper balance.

Types of Textile Research

Just what constitutes research and what does not, is always a good subject for argument. The term "research" has come to be very loosely used, and may indicate anything from simple trouble-shooting to the labor required in a highly specialized study. Dr. C. E. K. Mees, vice president and director of research of the Eastman Kodak Company, says that "research can be considered as organized effort to learn how to do things you will need to do when you can no longer do the things you do now." It would appear that such a definition is appropriate for the textile industry, as from all indications a plant that expects to continue using old-fashioned methods is going to find itself pushed out of the picture early in the post-war years.

Two General Types of Research

Permitting a sufficiently broad interpretation of the terms, all research may be placed in one of two categories, fundamental and applied research. Giles E. Hopkins, director of applied research for the Textile Research Institute, recently stated that "the concept of fundamental research . . . in general places the emphasis on the desire to determine basic principles which control the behavior of materials or energy in their various forms. . . . Applied research has the same relationship to fundamental research as engineering has to pure science. Essentially it is the step between the revelation of the principle as turned up by fundamental research and the application of this principle to answer known demands."

In many cases a good piece of applied research may be accomplished without a very complete knowledge of the fundamental principle involved. If we had had to wait, for example, until we knew all about the phenomena that would take place within a carding

machine before we could apply the principles to cotton, the human race would still be tapping its collective foot, waiting for the card to be invented. The fact is, we do not know yet exactly how the fibers behave in a card, but some 80,000 of the machines are in operation in this country every day! In other words, there is still a place for the empirical, or "trial-and-error" approach to research, although the efficiency of this method may well be questioned in many cases.

Because of the experience and mental make-up of our staff, and in view of the facilities available at the State College Textile School, we can expect to contribute more by confining our activities primarily to the field of applied research. There may be cases in which some work of a fundamental nature may be conducted, as for example the measurements of the physical characteristics of fibers under different degrees of relative humidity and temperature. But in general the greatest returns from research can be expected in a textile school if the program centers around the application of principles to commercial production, whether it be in bleaching, dyeing and finishing, or in the spinning and weaving or knitting of various materials.

There is one point that I should like to emphasize with respect to research of all kinds, and that is the time element involved. There are those in positions of authority in the textile industry who firmly believe that a month is ample to set up any research project and put it into action, and that, say, on every second Monday the project should produce a new, revolutionary and money-saving finding or it isn't worth the money spent on it. To those men I would call attention to Dr. Mees' statement, appearing in TEXTILE RESEARCH for December, 1942, that "It will take a new laboratory five years to find its feet. . . . And it will require at least ten years before the research laboratory pays profits." The actual time required for either, of course, will be determined by many factors, but the point is that successful research cannot be conducted with one eye on the clock and the other on the calendar. The supporters of research must have faith in the staff and its objectives, and they must expect that many blind alleys will be traversed before anything of outstanding value can be accomplished.

Additional Advantages for Research at State College

It is an elementary but frequently-overlooked fact that before embarking upon a research project it is well to spend a sufficient amount of time to survey the field and determine what has been done previously on the subject. To do this quickly and thoroughly requires adequate library facilities. At State College we expect very shortly to have an excellent textile library in our School under the supervision of a trained librarian, and of course we shall make full use of the library in our research.

We enjoy another advantage at State College which already has proven very valuable to the research we are doing. There are outstanding chemists, physicists, engineers, agricultural scientists, mathematicians and statisticians on the college faculty who are available at all times for consultation and cooperation, and assistance is being given to us by these specialists whenever the need arises. In fact, the administration is fostering the fullest cooperation among

the different units particularly with reference to research, and the value of such cooperation cannot be assigned too high a value.

In planning our research program we have high hopes of establishing a department of textile research, with a staff of trained people working under the direction of an outstanding scientist.

Financing Research at a Textile School

Many relatively small research projects can be managed at a textile school without the need for any additional funds whatever. It is our policy to carry out investigations of a limited scope for the mills of the State upon request and without charge. If the project requires considerable time and some new equipment and materials, it frequently is possible to undertake such an investigation for a reasonable fee, which merely covers the additional expenses. In some cases a research organization may sponsor a project at a school, the funds being obtained from its members. In other instances in which war requirements of the industry necessitate research, the Government may request and finance research in the schools. At State College, for example, we are currently conducting some research on cotton carding under the sponsorship and general technical guidance of the Textile Research Institute. In this case the work is being financed by the Government because it touches upon a vital production problem.

I hope that I have been able to give some insight into the relationship of the textile schools to textile research in general, and to the program of research at the State College Textile School in particular. If we boil down and concentrate the stream of our discussion, we may say, first, that the schools can, at least in some cases, contribute to research laboratories and to the industry by giving students a background of knowledge of textile research; and second, that with a suitable personnel and equipment at hand, the schools can contribute a great deal to the industry by conducting applied research in their own laboratories.

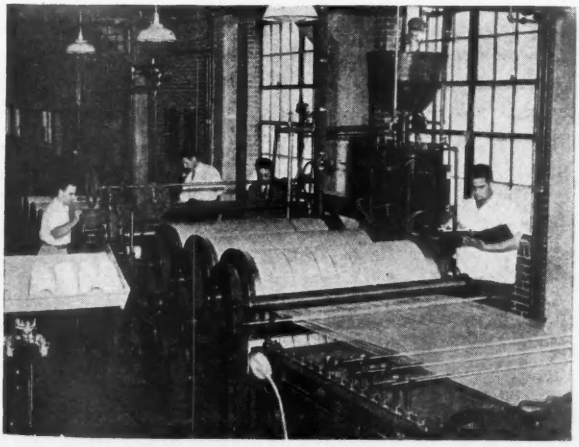


Fig. 3. Students conducting experiment on yarn slasher at N. C. State College Textile School.

Post-War Competition in Textiles Will Demand Dual Research Program in

Economics and Technique

By DOUGLAS G. WOOLF

Among the many popular misconceptions of research is that its zone lies in the field of technique alone. The term itself arouses in too many people's minds an automatic vision of a man with a test tube or a man peering into a microscope, period. So long as research deals with causes and methods having a bearing on production, the average man is satisfied. But let it veer into the zone of distribution or of economics, and many who hold this narrow view of research become almost scandalized. They feel let down: as if someone had taken their religion and debased it.

That the above is by no means an uncommon state of mind, I can testify out of my experience over a good many years. Again and again, I have heard people question whether economic research had any part in a research organization.

All of this would be almost humorous, if it were not also dangerous.

There may have been a time—and in fact there probably was—in the history of textile manufacture, let us say, when it was reasonably safe to ignore economics and concentrate on technique. That these periods were short-lived, however, is shown by any even casual study of textile history.

That the last quarter century, with the exception of the war years, has been a period when economics absolutely dominated the textile problem, is a self-evident fact. That the next quarter century will present even more critical economic problems to the textile manufacturer than has the last quarter century is, we believe, equally self-evident.

Unfortunately, many industrialists are blinding themselves, unconsciously, to this fact. Some men active in almost all the types of industries have practically hypnotized themselves into a belief that we have at last found the key to perpetual prosperity. Just how this idea has gained ground is beyond our comprehension. Certainly, it is based on the most flimsy of arguments. The idea is that we now have unlimited productive capacity and unlimited need; consequently these two supplementary forces are going to join hands and keep all the machines and all the men occupied at a profitable rate for both employers and employees. The same sort of argument was used after the last war. We had similar circumstances, although of course this time they are much more acute. And it is true that for a short while the world worked feverishly to fill the voids created by war. Then came the day of reckoning.

True, we have certain circumstances today which did not exist after the last

war and which might help to postpone, and possibly even avert, the day of reckoning. But those forces are not going to get into action on their own. They have to be utilized just as intelligently and just as carefully as any laws of physics or chemistry which bear upon technical problems of industry.

Take Textiles, for Example

If the above is true in industry generally, it is particularly important in connection with textile manufacture. Here we have an industry which is going to be beset by all kinds of competition. There will be the competition from within: the adjustment as among different types of fibers and different types of equipment, all facing new additions to these competitors constantly. There will be the competition from other industries for textile markets; what the paper industry has done, other industries can do and can be counted on to try. Also, there will be competition from other industries for an increasing share of the consumer's dollar; manufacturers of electric refrigerators, radios, automobiles, air-conditioning equipment, and what-have-you, will have a pretty good sales story to offer to people who during the dark war years could find something to wear but couldn't get a new ice box or a new automobile under any circumstances whatever. There will be competition from other nations: from those in which the awakening of an industrial consciousness finds as usual its first expression in textile manufacture, and those which for political reasons will manipulate their textile exports, by way of Government subsidies.

There will be international competition in connection with wages, and in connection with prices of raw material—both fair targets, also, for political manipulation.

What This Means in Research

Facing an unknown future in which the above are only a few of the possibilities, in fact probabilities, does it make sense to exclude economics from textile research? On the contrary, isn't it absolutely essential that economics be given a place of equal importance with technique in any research program? Also, isn't it necessary that the two be synchronized, so that the results of research in technique on the one hand and economics on the other may be examined realistically in the light of limitations and probabilities in the other field?

At least that is the attitude of the Textile Research Institute, Inc. It regards economic research as one of the major zones of activity for such an organization. It is now laying its plans for a study of the probable competitive positions of the American textile industry in the post-war era. The Textile Foundation authorized this study and asked the Textile Research Institute, Inc., to administer it.

The results of this research, as of all economic research to be conducted by the Institute, will be presented in a factual form to the industry. The Institute will make no attempt to influence the actions of the industry as a whole or individuals in it. Its activity will be based on the theory that the first requirement in economics, as in technique, is the facts. It will make every effort to dig out the right facts to give to the industry. It takes it for granted that textile manufacturers, as intelligent human beings, will attempt to use facts as a basis for formulation of their operating policies.

RESEARCH ACTIVITIES

The appointment of Dr. Hugh S. Taylor to succeed Dr. Luther P. Eisenhart as Dean of the Graduate School of Princeton University to take effect July, 1945, has been announced by Dr. Harold W. Dodds, president of the University. Dr. Taylor, who will be the fourth dean of the Graduate School, is internationally known as an authority in physical chemistry and has won many honors for research in catalysis. A native of England, he is now David B. Jones Professor of Chemistry at Princeton. Dr. Taylor is a member of the Textile Foundation scientific advisory committee which also serves as the fundamental research committee of the Textile Research Institute, Inc.

The Industrial Fiber Society will hold its fall meeting Friday and Saturday, September 22 and 23 at La Grange, Ga. The meetings will be held at the Callaway Auditorium. Opportunity will be provided for those present to visit the Callaway Mills and the Callaway Institute in La Grange.

Annual Intersectional Contest of the American Association of Textile Chemists and Colorists will be held Friday evening, October 13th, in connection with the annual convention of the Association which is to be held October 12–14, Hotel Claridge, Atlantic City, N. J.

"Essential Cotton Textiles," a 30 page booklet issued by The Cotton Textile Institute, Inc., Box 151, Clemson, S. C., and the National Cotton Council, Memphis, Tenn., describes pictorially many of the war applications for cotton textiles. Also illustrated are some of the essential industrial and civilian uses.

The first annual electronics conference will be held at the Medinah Club of Chicago, 505 N. Michigan Ave., Chicago, Ill., on October 5–7, 1944. The widespread use of electronic developments in the furtherance of this nation's war effort makes this conference of unusual timeliness. Emphasis will be placed on the engineering aspects of electronics in the conference which is described as a "national forum for electronic developments and their engineering applications." The conference is sponsored jointly by the Illinois Institute of Technology, Northwestern University; the Chicago Section of the Institute of Radio Engineers; and the Chicago Section of the American Institute of Electrical Engineers.

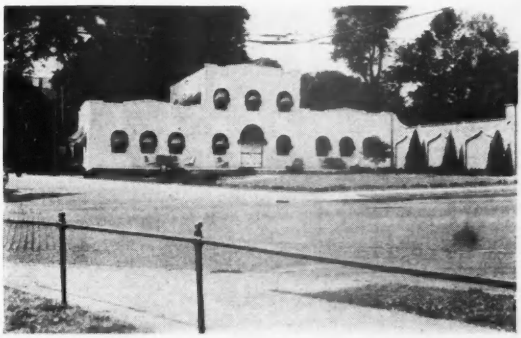
North Star Woolen Mill Company of Minneapolis, Minn., and the Lima Woolen Mills Company of Lima, Ohio, have appointed Raymond K. Flege as director of research for both mills. Mr. Flege, a chemical engineer, holding graduate degrees from both the University of Kentucky and the Massachusetts Institute of Technology, was a research associate at M.I.T. from 1931 to 1932, and with the Bauer and Black Division of the Kendall Company, as a research chemist, from 1932 to 1936. He then served as a chemical engineer

with the Kendall Mills, Finishing Division of the Kendall Company, from 1936 until he became associated with North Star.

A process for utilizing a Plaskon, Bakelite or other resin on fur fibers on the pelt to give the fur the properties of the plastic, has been invented by Jose B. Calva of St. Paul, Minn. (*Plastics News Letter*, July 3, 1944). The process uses the fiber as a reagent to get a chemical reaction between the amines in the fiber and the aldehydes in the resins, with the result that sheep pelts can be made to resemble beaver or seal (when the pelt is shorn), or fox or lynx (using unshorn pelts). It is said to be applicable to other fibers for industrial use, such as bristles, both natural and synthetic. At present 1,500 plastics sheepskins a day are being turned into beaver for aviators' suits. *Business Week*, June 24, has a brief article on the invention.

Owens-Corning Fiberglas Corp. is producing a new textile combining glass and asbestos fibers which currently is confined to war uses, in cord form in shroud lines, on military flares, and in fabrics for gun boots and tailwheel boots on military planes. The glass-asbestos textile is intended for applications requiring high strength combined with light weight, high abrasion resistance, and resistance to high temperatures and corrosive fumes. The high strength of the material is primarily due to the glass, while the asbestos increases abrasion resistance.

The new research laboratory of S. Stroock & Company occupies the second floor of a recently completed building adjacent to the company's mill in Newburgh, N. Y. The first floor has been equipped as a recreation center for Stroock employees. The building was formally opened July 8 by a gathering attended by Sylvan Stroock, president of the company, Mrs. Elsie Murphy, executive vice president, and other company officials and employees from the New York and Newburgh organizations. The laboratory under the direction of Dr. A. C. Whitford is being completely equipped with the most up-to-date apparatus for conducting research.



Research laboratory and recreation center, S. Stroock & Co., Newburgh, N. Y.

RESEARCH AIDS

Surface Chemistry*

(Reviewed by Carl M. Conrad.)

This book comprises a collection of ten papers based on a symposium held in September, 1941, in celebration of the fiftieth anniversary of the founding of the University of Chicago and in commemoration of the twenty-fifth anniversary of the first contribution of Dr. William D. Harkins in the field of surface chemistry. The articles represent the individual or joint efforts of 15 authors, all outstanding authorities in their field, and are edited by Dr. Forest Ray Moulton, and published as No. 21 of the American Association for Advancement of Science. While reporting original investigations, the articles are presented, for the most part in expository or interpretative style. Most excellent and extensive references to the literature are given. Mathematical treatments are used freely and the reader's aptness in this field is taken for granted. In one or two cases the connection between the subject presented and surface chemistry is not too obvious, as for example the article dealing with the problem of elasticity of rubber and rubberlike materials. The reviewer observed that the volume might have been entitled "Surface Physics" with almost equal justification as with the title used. Considerable attention is given to methodology, particularly to special refinements. The collection should be a most valuable asset for investigators specializing in this field.

* SURFACE CHEMISTRY, edited by F. R. Moulton. *Science Press Printing Co., Lancaster, Pa. 160 pp. Price $3.25.*

Soybean Chemistry and Technology*

Soybean Chemistry and Technology is an authoritative and timely book that will meet the demand for up-to-date facts covering the chemical and physical aspects of the versatile soybean. Written by scientists of wide experience in the field the composition and properties of the soybean, its constituents and products, are dealt with in detail. Largely based on experiments of the authors, it presents in addition a valuable summary of numerous research contributions which have appeared up to 1943 in technical literature throughout the world.

* SOYBEAN CHEMISTRY AND TECHNOLOGY. Klare S. Markley and Warren H. Goss. *Chemical Publishing Company, Inc., 26 Court St., Brooklyn 2, N. Y. 261 pp. Price $3.50.*

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Marketing Research Agencies

A survey and directory of marketing research agencies in New York City has just been published by the Bureau of Business Research of The City College School of Business, 17 Lexington Avenue, New York, N. Y. Price $1.00. It contains a list of the agencies which do independent commercial research work in New York City, and indicates the line of activity of each agency, whether it specializes in a particular field of research such as radio research, drugs, textiles, advertising media, economic research, personnel and industrial relations, editorial research, management research, etc., or whether it offers a general line of marketing research.

Both Government and Industry Would Profit by Proposed

Statistical Program

Statistics bear somewhat the same relationship to some forms of economic research that test methods and specifications bear to some forms of technical research. They are an important tool. As such they are of significance to every organization interested in economic research. At present a program is developing to insure the progress made in the gathering of statistics during the war by the Government agencies. This program was outlined recently and sent to interested groups by Jesse Jones, Secretary of Commerce. The outline follows.

* * *

During the war the statistical facilities of the Department of Commerce have been largely converted to the compilation of information specifically related to the immediate needs of the war agencies. Considerable time is required to develop a statistical program and to do all the work preliminary to operating such a program. The Department has already begun the necessary developmental work to reestablish the flow of statistical information to business and to provide needed facts for postwar operations as soon as military security permits.

Such a statistical program must provide both Government and business with the factual information on production and distribution needed not only during the difficult transition from war to civilian production, but also for efficient continuing operation. To meet these needs, the Department has under development the following program:

1. From an immediate standpoint, cooperation with the war agencies, particularly the War Production Board, in analyzing and sifting the large body of wartime administrative and statistical data with a view to extracting and releasing as much of it as appears useful to business and industry.

2. From a longer range standpoint, conducting the biennial Census of Manufactures, and the Censuses of Business and Mineral Industries (along the lines of the 1940 Census) in 1946 covering calendar year 1945. A sample survey of population designed to cover the major wartime shifts and the quinquennial Census of Agriculture are also planned.

3. For both immediate and longer range purposes, developing a current statistics program to provide government and industry with the information needed on current changes in industrial operations. This program will provide essential information with a minimum expenditure of effort on the part of both industry and Government.

Immediate Projects

The "Facts for Industry" series through which information collected by the War Production Board, except in a few special cases, is being released through the Department. This series now covers wartime production for a

wide variety of commodities in the metals, chemicals, textiles, wood products, machinery and equipment industries. This series is being expanded as industry makes its needs known and military security permits.

In addition, a series of economic reports is being prepared by the Bureau of Foreign and Domestic Commerce and will be released publicly as rapidly as possible. These are developed along industry lines and will draw upon and coordinate data from the industrial statistics program, as well as from other sources. They present an analytical interpretation of the economic position of each industry covered in such a way that the Department will be simultaneously performing its function as industrial consultant to Government and economic consultant to industry. Such a report, covering the paper and pulp industries, is being released currently, and is indicative of the objective sought.

Furthermore, some of the restrictions governing the release of foreign trade statistics have been removed so as to permit the publication of complete United States import and export statistics through 1941. With the exception of certain strategic items, information on trade with Latin America will be published with a lag of 12 months; trade with Canada and Mexico 6 months late, and complete commodity statistics 12 months late. Additional current statistics on the foreign trade of the United States will be made available as rapidly as consistent with requirements of military security.

For Post-War Guidance

To provide a reasonably complete inventory of the impact of the war on the nation's human and material resources so that the essential facts are available to business and industry for scheduling postwar operations, the Department of Commerce is planning to conduct in fiscal 1946 the Biennial Census of Manufactures, a Census of Mineral Industries (along the lines of the 1940 Census), a Census of Business, and a sample Census of Population. These census activities, together with the quinquennial Census of Agriculture to be taken in 1945, will provide business and industry with basic facts for planning future operations.

Efforts to increase the usefulness of Census of Manufactures data to both Government and business have been intensified, particularly in the direction of increasing the amount of information available on specific commodities and the consumption of materials. By redesigning and increasing the number of tabulations, the amount of information useful to market analysts and management engineers will be increased with little or no additional burden to industry.

Facts on Distribution

Information on distribution for both consumer and industrial markets will be particularly important in the transition period and in postwar operations for some time to come. At the present time there is no knowledge available on the over-all impact of the war on our channels of distribution. To provide this badly needed information so that the essential facts are available to business as a basis for returning to normal activity, the Department is planning to request funds to take a Census of Business in 1946 covering 1945 activities, including wholesale and retail trade, services, and construction.

To complete the outline of the program of benchmark statistics for business and industry, it is planned to conduct a sample census of population. The war has occasioned great shifts in population within the nation which has considerably altered the distribution of population as reported in the 1940 Census. The population shifts which have occurred have involved not only great increases in war production centers but considerable decreases in other sections of the country and in the characteristics of the inhabitants of many areas. Information about the changes in both the distribution and characteristics of the population is vital for consumer market analysis. The Bureau of the Census has, therefore, prepared plans for obtaining such information at minimum cost through the use of sampling techniques. The cross section survey of population planned will provide statistics for the nation as a whole, and for each State by urban and rural areas, for each city of 100,000 or more inhabitants, and for smaller cities by city size groups.

A Current Reporting System

Finally, a system of current reports covering the more important commodities and segments of the industrial economy is essential if we are to understand the significance of fluctuations in the current scene.

The Department is marshaling its resources with a view to designing, with the cooperation of other Government agencies, a current reporting system which will supply the minimum information needed by both Government and industry. Advising the Department on the types of information needed, the commodities to be covered, and on the analyses needed to make such information most useful are technical experts and commodity specialists representing the Labor Department, Agriculture Department, Treasury Department, Interior Department, U. S. Tariff Commission, Federal Reserve System, Social Security Board, War Production Board, and the Bureau of the Budget. These groups will assist also in relating the Department's statistical program to the data collected by other agencies in special fields.

In order that no unnecessary burden is placed upon industry in supplying the basic information for such a program, no stone is being left unturned in making full use of previously collected data and the most advanced statistical techniques for designing questionnaires and for sampling where such methods are indicated. Just how extensive this current program needs to be will depend in a large part on industry's needs for specific, detailed data for its own operations. The analytical resources of the Department will also be used in seeing that information so collected is processed so that it can be made available in a form that will insure its maximum usefulness.

In developing this statistics program the Department expects to obtain the advice and counsel of industry and business, as well as that of interested agencies of Government. During the developmental stages of the program, individuals from the business community and from Government will be called upon to advise with our commodity specialists regarding technical problems. When these technical problems are out of the way and individual programs begin to take shape, opinions and suggestions of those concerned or interested in the program will be solicited.

Research That Has Furnished the Background for Current Practice is Described in

The Story of Coated Fabrics

II—Rubber and Pyroxylin Coatings

By FRANCIS J. TUTTLE
Research Chemist, R. B. H. Dispersions, Inc., Bound Brook, N. J.

The evolution of methods for the manufacture of coated fabrics has followed a sequence as the three main classes of raw materials became available, commencing with vegetable oils and leading to the modern synthetic resins. Much of the current technique follows closely some of the practices described in these articles. Part 1, which described the development of oil cloth, appeared in July TEXTILE RESEARCH. *The following article concludes the series.*

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Rubber-Coated Fabrics

Rubber first became known in Europe about 1525 being mentioned at that time in the writings of d'Anghiera, a Spanish traveler in Mexico. Later in the century Portuguese explorers introduced into Europe watertight bags made from this material in Brazil. In 1615, Jean de Torquemada stated that the Spanish conquerors in Mexico used the latex for painting linen coats for protection against rain. He noted that water did not penetrate the fabric, but he observed that sunlight had a deteriorating effect on the product. The French Academy of Science sent a scientific expedition to Peru about 1736. One of the members, De la Condamine, sent back to France samples of the material which natives called "caoutchouc." He described the native method of obtaining the gum from the latex, making waterproof garments by smearing fabric with the milky latex and drying the coated cloth over a fire. The interest of French scientists was excited by this report, and France remained in the forefront in the study of this new material for the rest of the century.

A chemist, Macquer, reported that ether was a good solvent for the caoutchou. Herissant, a physician, suggested turpentine as a solvent. An Italian engineer, Fabroni by name, reported in 1791 that petroleum would dissolve the rubber. Joseph Priestly, famous for his work with oxygen, found the gummy caoutchouc very useful for erasing pencil marks and suggested the name "rubber."

It was about the turn of the 18th century that interest in the use of rubber spread to England. Thomas Hancock, a former mechanical engineer, became interested in rubber, and in 1819 he experimented with solutions of rubber in turpentine but was discouraged by the bad odor and very poor ageing of the product. Neverthe-

less, he started a modest business cutting threads and bands from rubber blocks. These he sold for use as garters and armbands. There was much scrap left from this operation and Hancock devised a machine for the purpose of shredding and pressing a new block of rubber from the scrap. The shredder had a capacity of about two ounces and was made of hardwood parts with an interior space that could be tightly closed. Centered in this space was a wood-covered shaft turned by an outside crank. The surfaces of the interior and the shaft were studded with iron spikes to help shred the rubber. The full power of one man was required to turn the shaft when the interior was loaded with rubber scrap. Hancock, in his first attempt, loaded the device and turned the crank intermittently for almost an hour. When he opened the chamber he found, to his surprise, that instead of the expected shreds, he had produced a solid mass of masticated rubber.

Encouraged by this result he built a larger machine which would masticate one pound of scrap and required the strength of several men to turn the shaft. In 1820 he built a horse mill to operate a still larger masticator. Shortly thereafter he added a pair of iron rollers to his plant equipment for sheeting out the masticated rubber. He did not patent either device but, pledging his men to secrecy, he kept knowledge of the machines from his competitors for twelve years.

Meanwhile, in Glasgow, a Scotch chemist, Charles Macintosh, contracted with the Glasgow Gas Works to purchase their by-product naphtha for a number of years. He began making waterproof coats for the trade and in 1823 patented a new product which gave great impetus to the proofing industry. He first spread his rubber solution (10% rubber in the by-product naphtha) on a smooth marble slab. When the solvent was evaporated he sandwiched his thin sheet of sticky rubber between two pieces of cloth from which he then fashioned raincoats. By having his rubber between the cloth he eliminated the cause of much complaint against the rubber-surfaced raincoat of the time, which in a warm place or in summer weather became very sticky and soft.

About this time Hancock was experimenting with latex for use on fabric but the difficulty in importing the latex in a stable condition led him to abandon this venture. In 1825, the year Macintosh moved his business to Manchester, Hancock obtained a license from him to produce a double texture material. Hancock was then able to produce a cheaper and superior product because of his mastication process which allowed higher percentages of rubber to be dissolved in the same amount of solvent. In addition to raincoats, he began to make air-mattresses, pillows, and life preservers.

The well-known British explorer, Sir William Edward Parry, in his "Narrative of an Attempt to Reach the North Pole," published around 1827, paid tribute to the early waterproofing art. He reported that a rubberized canvas bag containing cocoa fell into the sea and was recovered without any salt water entering the bag. He writes; "of this invaluable manufacture which consists, I believe, in applying a solution of elastic gum or caoutchouc between two parts of canvas it is impossible to speak too highly. I know of no material which with an equal weight is equally durable and

water-tight—in the latter quality indeed, it is altogether perfect as long as the material lasts."

Hancock introduced the art to France in 1828. Here he installed his machinery at St. Denis for Rattier and Guibal. He also instructed them in the production and use of solutions for coating fabrics. Shortly thereafter he joined company with Macintosh and installed a machine at Manchester which would masticate 200 lbs. of rubber at one time.

In April, 1834, the upsetting of a candle on a piece of cloth being proofed caused the complete destruction of the Manchester Macintosh plant by fire. In the summer of 1838, the Manchester works was again destroyed by fire of unknown origin.

David Moseley & Sons, Ltd., began proofing in a modest way in 1833 and in 1840 S. W. Silver & Company started a small waterproofing business at Greenwich. In 1852 they moved to a new location, prospered and gradually a district was built up around them which became known as Silvertown.

Other manufacturers began operations, and by 1840 it was estimated that 3,000 to 4,000 square yards of fabric were produced per day. Around 1840, however, a general decline in the waterproofing trade resulted because of various undesirable properties of the product; these defects were not remedied until Goodyear's principles of vulcanization were generally appreciated and applied.

The combination of sulfur and rubber appears to have been used some years before the work of Goodyear. As we have seen, much of the work with rubber was performed by means of solutions. As early as 1831, a Samuel Guthrie of New York City is said to have dissolved sulfur in turpentine and used the mixture as a rubber solvent. What he did with the solution and what results he obtained are unknown. Friedrich Ludersdorff in 1832 published in Germany an observation to the effect that when rubber was dissolved in turpentine and 3% sulfur was added the film formed by evaporation was less tacky than when no sulfur was present. In 1839,

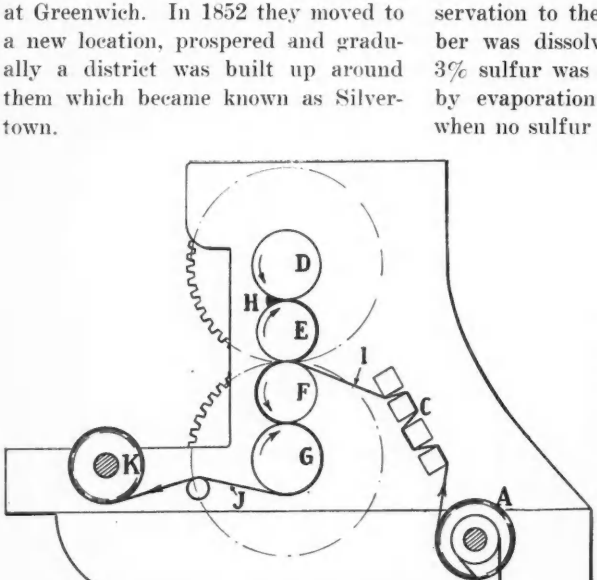

Fig. 9. The Chaffee Calender (1836).

The cloth *I* was led from the roll *A* under tension from weight brake *B* through tensioning and straightening bars *C* into the rollers *F* and *G*, which were hollow iron cylinders arranged for heating; the bank of rubber compound *H* was applied to the fabric between rollers *E* and *F*; the coated fabric *J* was rewound on *K*.

Nathaniel Hayward prepared a mixture of raw rubber with sulfur as an ingredient.

It should be noted however that all the early rubber workers missed the essential fact discovered by Goodyear in 1839 at New Haven. He was the first to recognize the profound changes resulting in rubber from a combination of time, temperature and sulfur. He not only noted the improved physical properties of the rubber such as increased strength along with the "metallizing" or drying of the surface, but he also recognized that its stability to heat and cold as well as resistance to atmosphere and wear were greatly improved by the proper balance of this combination.

In 1843, quite independent of Goodyear, Hancock obtained a patent in England for improving rubber properties by dipping strips of rubber into molten sulfur. A classically-minded friend of Hancock's, Brockedon by name, called the process "vulcanization." Shortly after this (1846), Parkes in Birmingham observed that vulcanization resulted when thin sheets of rubber were immersed in a solution of sulfur monochloride (S_2Cl_2).

Until 1836, the preparation of proofed goods was a slow, tedious process of applying rubber doughs by hand to the cloth. In that year Edwin M. Chaffee, then connected with the Roxbury India Rubber factory in Massachusetts patented a mill and calender. He claimed the following: improvement in the art of application of undissolves rubber to cloth; the coloring of the rubber without the use of solvents and his particular machinery for accomplishing both ends. The preparing machine, as he called it, consisted of a hollow cylinder 6 feet long and 27 inches in diameter adjacent to another cylinder of the same length but 18 inches in diameter. Both could be heated by steam to about 200° F. By the use of heat and pressure various pigments could be incorporated into the rubber by masticating it between the two rolls.

Chaffee's calendar or "covering machine" consisted of four hollow rolls placed in a frame one above the other and all 6 feet long. The top and bottom rolls were 18 inches in diameter, the two inside rolls 12 inches in diameter and all could be heated with steam. The milled rubber was sheeted

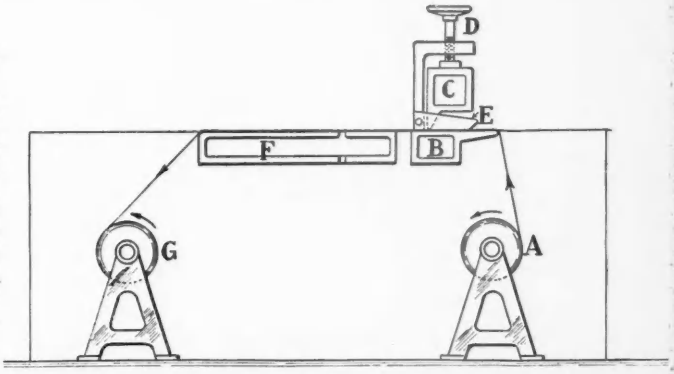

Fig. 10. The Hancock Spreader (1837).

The cloth from roll *A* passed over steam-heated bed-plate *B* under knife *C;* the knife was blunt and adjustable vertically by *D; E* was a guide to prevent coating dough from spreading beyond cloth edges; *F* was a steam-heated drying table; *G* was the rewind roll for finished fabric.

between the two top rolls, cloth and rubber were combined between the two middle rolls and smoothing was completed between the two bottom cylinders. Chaffe's calendar weighed 30 tons and was called the "Mammoth." The cylinders for it were imported from England and the total cost amounted to some $30,000. The Roxbury Co. purchased the inventor's entire interest in the "Mammoth." In 1843, at a public auction a Mr. Haskins purchased it outright for $525 together with the patent for an additional $1.50. A year later Haskins sold the machine to Goodyear who after a short time resold it to the Naugatuck India Rubber Co.

Meanwhile in England (1837), Hancock obtained a patent for a machine to spread his rubber solutions on cloth. The standard spreader, in use today, is based on the general principles disclosed by Hancock. This consists essentially of a steel spreader roll covered by a layer of hard rubber mounted between two side frames of cast iron with cross pieces to hold them rigidly in place. Mounted above this roll and ground very closely to the roll is a knife adjustable vertically. Brackets set on the frame carry the uncoated roll of cloth which is conducted between the hard rubber roll and the spreader knife. The dough is applied by adjusting the space between the knife and roll and the coating is dried by conducting the cloth over a hot table formed by steam heated pipes.

Departures from the standard type were numerous. The Frankenstein-Lyst spreader was very similar to the standard type with one spreader roll but instead of one knife, four were used with each successive one set closer to the fabric. The Rowley-Walmsley machine was a double deck type. Two rolls of cloth could be coated simultaneously by one operator. By a simple reversing device the front knives are lifted, the back knives let down and both fabrics were recoated from the other end of the machine eliminating the necessity of winding back the coated cloth. The Coulter spreader was a single deck variation of the double ended machine. The Salisbury spreader was designed so that two or more coats could be spread in one op-

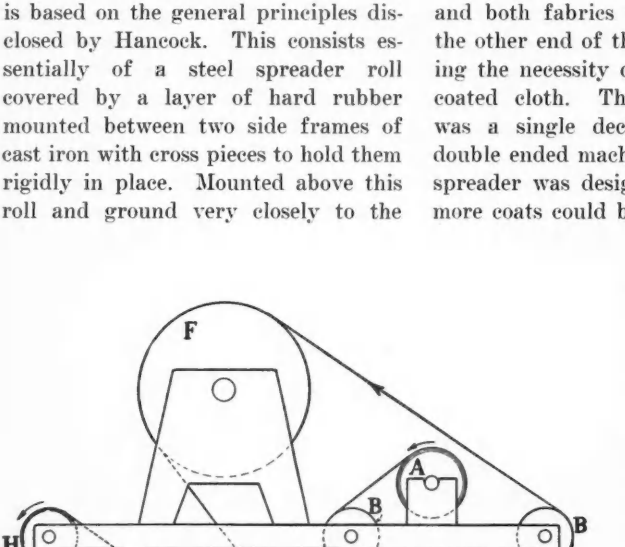

Fig. 11. An early device for cold-curing rubber fabrics.

A was cloth-take-off roll; rollers *B* and depressing guide *C* maintained cloth tension; *D–D* were two fountains containing carbon disulphide solution of sulfur chloride which was applied to rubber coated surface of fabric by contact with rollers *E–E*; heating drum *F* evaporated solvent; *G* was a breaking roll conducting cured cloth to rewind *H*.

eration with sufficient drying between each coat. The Birley-Macintosh spreader was equipped with an endless chain which held clips that were fastened to the cloth while it was drying thus maintaining the cloth under tension across its width. In the Hawkins spreader the knife was replaced by a variable speed roller on which the pressure could be adjusted in the vertical direction.

The processes developed during the 1850s have changed little in principle and are the basis of modern rubber proofing and coating. These processes include mill compounding, churning of the compound in a suitable solvent, spreading the dough, vulcanization of the coated cloth and in some cases starching, varnishing and embossing the proofed fabric. Solarization or sun cure was widespread until the turn of the century. Tables were set out of doors so that the sun's rays fell directly on the rubber surface. It was gradually replaced by either dry heat, cold cure or vapor vulcanization, the method chosen depending on the type of product.

The significant changes that have taken place were not so much in processing as in compounding. As early as 1840 Andrew Ure described the effect of lime in "drying" rubber. Goodyear also noted the effect of metallic oxides in accelerating his sulfur reaction. In 1884 N. S. White used aniline to give brilliance of color to his pigmented rubber but he did not seem to observe any effect of this material on the rate of vulcanization. Dr. Weber in 1904 found that he could get better vulcanizates with low grade rubbers by using stearic acid to overcome the deficiency in acid content of these rubbers. After considerable research George Oenslager in 1906, established that aniline and other organic substances of nitrogenous nature profoundly accelerated the rate of vulcanization and greatly improved the physical properties of the resulting vulcanizate. As a practical result of this work, manufacturers could vulcanize in from one-third to one-fifth the time formerly required and obtain at the same time superior products.

Pyroxylin-Coated Fabrics

In 1833, Braconnot, a French chemist who had done considerable research with cellulose, was led, in the course of his investigation, to try the reaction of anhydrous nitric acid on various polysaccharides, including cotton, linen, and wood shavings. He obtained

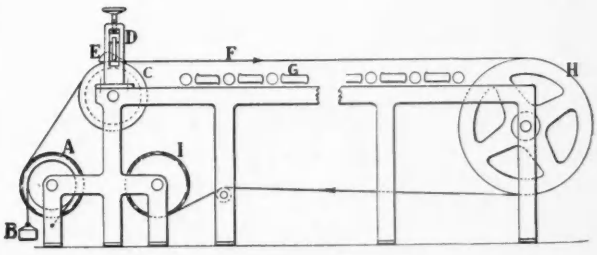

Fig. 12. Standard Spreader.

A is the cloth take-off roll with braking weight *B*; *C* is the spreader roll of rubber-surfaced steel; *D* is the adjustable coating knife; *E* is a lateral guide to keep the dough on the fabric; *F* is the coated fabric; *G* are steam coils for drying the coated cloth; revolving drum *H* is also heated; *I* is the rewind roll.

highly flammable neutral powders soluble in concentrated acetic acid. These acid solutions deposited tough, varnish-like films which were as clear as glass. This phase of his work was published in Germany and drew little attention, aside from an addendum by Justus von Liebig to the effect that Braconnot's products contained nitrogen and detonated weakly on heating.

Early in 1845 a chemistry professor at the University of Basel, Christian Fredrich Schonbein, brewed a particularly unsavory concoction on his kitchen stove while his wife was in town marketing. His pot contained, among other things, nitric and sulfuric acids. Suddenly, with much fuming and bubbling, it boiled over and the acids spilled on the clean kitchen floor. The professor was stricken with thoughts of his wife's impending return and in his haste to repair the damage from his mishap, he seized a cotton apron, mopped up the spilled acids, and generously doused the apron in the ever-handy pail of water. As his excitement subsided, he proceeded to salvage the soiled apron by drying it over the hot stove. Shortly there was a sudden puff and the apron vanished, leaving neither smoke nor odor.

Thus legend describes the epoch-making discovery of guncotton.

To lend credence to the story it should be remembered that the nitration of cellulose, using sulfuric acid as a catalyst, proceeds very rapidly, nitrogen contents approaching 12% having been obtained in less than three minutes under some conditions.

Further work with the nitration of cellulose, which followed these first discoveries, soon disclosed that the lower nitrates (pyroxylin) were soluble in an ether-alcohol mixture. This discovery is attributed to a medical student in Boston, Maynard, who proposed the use of these solutions, which he called "collodion," as a surgical dressing.

The first record of the application of nitrocellulose to fabrics appeared in 1855, Cornides (Eng. Pat. 745) suggested that paper and fabrics could be coated with explosive cotton, dissolved in wood spirit, pyroxylin spirit or alcoholized ether. Optional constituents in these formulas were india rubber, gums, resins and bitumen. Shortly thereafter Parkes (Eng. Pat. 2359) found that fabrics could be waterproofed by means of a solution of guncotton in vegetable naphtha, alcohol and methylated or other ethers.

The next improvement in the art of compounding was suggested in 1858 by Rollason (Eng. Pat. 2849). According to his patent, fabrics would be waterproofed with a composition prepared by dissolving pyroxylin in its known solvents to which is added a vegetable oil, such as castor oil.

In 1874, George and Robert Gray (U. S. Pat. 149,216) proposed the use of a new compound for waterproofing textile fabrics. This formula consisted of collodion and a solution of india rubber in castor oil. Miscibility was attained, it was claimed, by adding ether. A typical formula, suggested by them, was as follows:

India rubber	1 oz.
Collodion	2 oz.
Zinc white	4 oz.
Castor oil	8 oz.
Zinc sulphate	1 oz.

Optional constituents might consist of French chalk, aniline dyes, and pigments.

In 1882 John H. Stevens patented the use of a number of esters, among them amyl acetate, as active pyroxylin solvents. The first mention of "artificial leather" occurs in a patent granted to William Wilson and J. Storey (U. S. Pat. 352,726) in 1886. A formula suggested by them consisted of:

Amyl acetate	600 parts
Nitrocellulose	200 parts
Castor oil	300 parts
Pigment	Optional

While a great store of experience in the art of using nitrocellulose for coating fabrics was accumulated, no attempt was made to exploit this knowledge on anything approaching a commercial scale before 1890. There were several reasons for this. In the early years of the development of the manufacture of nitrocellulose the insoluble guncotton was almost exclusively produced. A number of disastrous explosions occurred which were caused by the instability of the product. The use of unstable nitrocellulose was found to result in artificial leather which had inadequate ageing properties, developing cracks and brittleness. The development of stabilizers to be added to the nitrocellulose, to combine with the acidic products of decomposition and so prolong its life, has resulted in the gradual increase in the quality of the material. Use of these

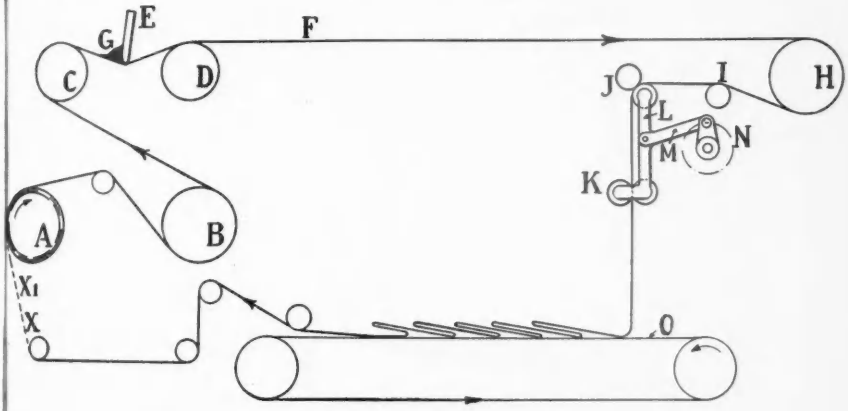

Fig. 13. The Mann Coating Machine.

This is a type which has had wide use among pyroxylin coaters. It spreads multiple coats while cloth travels in one direction in endless-chain fashion. The cloth is first passed through the machine with an attached leader which is threaded through the machine. Instead of winding the fabric after the first coat, it is folded on a traveling table which conducts the folds back toward the front of the machine. The lapping is continued until all the fabric is unwound from batch roll *A* at which time the end *X* has reached the front of the machine again. Here it is joined to uncoated end *X*, and the cloth is re-run the desired number of times; *B* is a brake drum; *C* and *D* are guide rollers which maintain the cloth *F* under uniform tension; the pyroxylin compound is spread at *G* by knife *E* which is adjustable both horizontally and vertically; *H* is a brake drum conducting cloth over idler *I* and between tension rolls *J*; from this point the cloth runs through a lapping mechanism which consists of two rolls *K* on the lower end of the swinging arm *L* which is rocked back and forth by connecting rod *M* and crank *N*; the fabric is thus laid in loose folds upon traveling apron *O*. By this arrangement a considerably longer yardage of cloth can be handled than would be possible by a straight run through.

stabilizers supplemented the purification treatment in the manufacture of the product. Without these the nitrocellulose explosive and lacquer industries were doomed to failure.

The lack of sufficient quantities of suitable solvents for applying nitrocellulose to cloth also greatly handicapped the commercial exploitation of this art. Likewise, the scarcity of plasticizers which were suitable for flexible coatings had much to do with retarding the growth of the industry.

In 1889, the Frederick Crane Chemical Company, which was producing photographic films, began experiments with the hope of producing artificial leather from pyroxylin. In 1890 a small plant was erected at Short Hills, New Jersey, and production was started on the basis of patents granted to Walter D. Field, of Millburn, New Jersey (U. S. Pat. 478,955) and Frederick Crane (Eng. Pat. 3,345). The composition patented by Crane was as follows:

Amyl acetate	7 gal.
Petroleum naphtha	3 gal.
Pyroxylin	15 lb.
Sulfuretted oil	30–40 lb.
Pigment	2–3 lb.

The principal claim of Field's patent is the softening oil used in the pyroxylin composition and a method of preparing it. Drying oils such as linseed were not deemed suitable, but nondrying oils containing unsaturated fatty acids, such as cottonseed or olive oils, were well adapted. By aerating the oil at 200° C. until the required viscosity was reached, and dissolving it in a blend of amyl acetate and petroleum naphtha, it could be combined with the nitrocellulose.

Just before the merger of the Crane and the Celluloid Varnish Company, in 1891, under the name Celluloid Zapon Company, it was decided to keep the "artificial leather" department apart from the new organization. The Tannette Manufacturing Company was thereupon organized and a plant erected at Bloomfield, New Jersey. Early in the twentieth century Tannette combined with the Evans Artificial Leather Company and later both merged with the Boston Artificial Leather Company. About this time the name of the product was changed from "Tannette" to "Moroccoline" and manufacture was transferred to Stamford, Connecticut, where it was produced by the Zapon Leather Cloth Company. The principal merit of the early product was its cheapness compared to leather, in spite of the fact that nitrocellulose at that time cost from $2 to $2.50 a pound. Since the coated fabric was produced in wide rolls, however, its use resulted in much less waste than was involved in the use of individually tanned hides. The sulfur-treated oils sometimes used as softeners, and the residual odor of the crude solvents that were used, gave the product an unpleasant odor which militated against its widespread use. This condition was aggravated by the development of rancidity in the semidrying vegetable oils used as softeners. These oils were often saturated either with oxygen or sulfur to obtain compatibility with the pyroxylin and to avoid the danger of spontaneous combustion in contact with the fabric.

Sale of the product under the description "artificial leather," also had much to do with the early prejudice against its use. By 1898, some progress had been made in overcoming part of the early resistance, and the bookbinding trade began to accept the

product on its merits. While there seem to have been several suppliers in the field by 1900, the only other company that is definitely known to have existed at that time was the Keratol Company, in Newark, which began business in 1898. By 1903, pyroxlin-coated fabrics were being used with success for furniture upholstery.

The passage of laws in 1906 permitting the sale of tax-free industrial denatured alcohol led to the adoption of a solvent blend containing alcohol, naphtha and ethyl acetate. It was soon discovered that the old oils used as softeners were no longer compatible in the new solvent blend and castor oil then became the standard plasticizer. During this transition period the pyroxylin coated fabrics used in the side curtains and folding tops of baby carriages proved to be so durable under constant flexing that their popularity greatly increased. Their introduction shortly thereafter into the automobile, in upholstery, side curtains and folding tops, pushed the industry into the "millions of yards" per year class. By 1909 the industry had so expanded that 11,000,000 square yards, worth $3,500,000, were produced in the United States in that twelve months.

In 1910 the DuPont Company started its "Fabrikoid" division for the production of all types of pyroxylin coated fabrics.

At the end of the First World War, the number of manufacturers had reached fourteen and the industry was established on a firm basis with a yearly dollar volume of close to $20,000,000 and a finished yardage of 38,000,000 square yards.

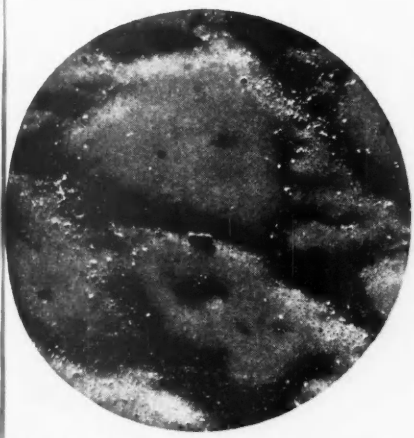

Fig. 14. Photomicrograph of surface of embossed nitrocellulose coated fabric (18 ×).

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ABSTRACTS

Members of Textile Research Institute, Inc., desiring complete copies of articles abstracted may obtain photostat copies at cost by addressing the Institute's office, 10 East 40 St., New York 16, N. Y. The price of a photostat negative is seldom more than 30 cents per page of original text.

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ANALYSIS: TESTING: LABORATORY METHODS

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Compression Meter

Compression meter for evaluating the compressibility and resilience of fabrics. Edwin C. Dreby. *Am. Dyestuff Reptr.* **10**, 199–204 (May 8, 1944).

A compression meter was devised for evaluating the compressibility of soft-finished, lightweight fabrics under pressures between 0.05 and 0.50 lbs. per square inch. The combined pressures of a liquid column and air under pressure are transmitted to the test specimen through a very thin, pliable membrane. Thus low pressures are distributed relatively uniformly over the test area. The results of measurements were compared with the hand, or feel, of the fabrics as judged by a group of textile experts. These comparisons indicated that the compressibility affects directly the feeling of "thickness." By combining the results of measurements of compressibility and flexibility, a measure of "fullness" was obtained. The compression meter can also be used to evaluate compressional resilience.

Oxygen Determination

Determination of oxygen in organic compounds. Philip J. Elving and W. B. Ligett. *Chem. Rev.* **34**, 129–56 (Apr. 1944).

The various methods proposed for the direct determination of oxygen in organic compounds are critically reviewed. The methods are classified as (a) those based on complete oxidation of the compound, (b) destructive chlorination, and (c) reduction methods. The first two are criticized as being time-consuming and inaccurate. The third class includes the important ter Meulen method and the simpler Schütze method. These two are reviewed in detail. The Schütze method, as modified by Unterzaucher and others, appears to be more promising for a variety of reasons. It is particularly interesting to carbohydrate chemists as good results are obtained with polyhydroxy alcohols where the ter Meulen method fails. The Schütze method involves thermal decomposition of the sample in a stream of nitrogen. The cracked products are led over carbon at 1000–1100° C resulting in the quantitative conversion of all oxygen to carbon monoxide. The carbon monoxide is then oxidized at room temperature with iodine pentoxide, yielding carbon dioxide and iodine which may be determined separately if desired. Inherently, the method is simple, accurate, and dependable.

Spectrophotometric Analysis

Spectrophotometric prediction of color of wool blends. E. I. Stearns and F. Noechel. *Am. Dyestuff Reptr.* **33**, 177–80 (Apr. 24, 1944).

A relation has been empirically found by the use of spectrophotometric data which permits the prediction of reflectance of mixtures of wool fibers from known reflectances of the components. The method disproves the fundamental assumption of additive reflectances as applied to the mixture of wool fibers but provides data for building special cams for the spectrophotometer that will give data linear in the additive function instead of linear in reflectance. Experiment shows that a straight line relation can be found for all wave-

lengths and for mixtures of colors other than black and white, as well as for wools varying in diameter. It seems remarkable, but a single empirical curve is sufficient as a basis for working out each combination of colors in which one is interested, regardless of wool diameters. A communication from the author states that for particles other than wool that show greater variation, the same relation does not hold, but that it is only necessary in that case to change the constant B. (See U. S. Patent 2,344,748.) The paper includes discussion of the advantages of using the spectrophotometer for improving mill matches which may vary because of variability of light, and mention is made of the use of single standardized artificial daylight lamps. Although the author does not include the information, it should be pointed out that there is wide practice in the textile industry of using color matching units which include two illuminants, one an artificial daylight, the other a yellow tungsten light. Use of such color matching units provides the color matcher with a sort of abridged spectrophotometer of two widely differing illuminants under which he can test his matches.

Cuprammonium Viscosity

Flow characteristics of dispersions of cotton and regenerated cellulose rayon fabrics in cuprammonium; their significance in fluidity calculations. Viola C. Jelinek. *Ind. Eng. Chem., Anal. Ed.* **16**, 172–8 (Mar. 1944).

Flow-pressure and fluidity-velocity gradient graphs were used to determine the flow properties of cuprammonium dispersions of cellulose fabrics and to evaluate the method of calculating the fluidity values. The limits of application of the kinetic energy and velocity gradient adjustments were determined for cellulose-cuprammonium dispersions under experimental conditions very similar to those recommended by the American Society for Testing Materials. The viscometer and buret consistometer were compared in the fluidity technique, the buret type instrument having the average over the single-volume viscometer type for the determination of complex cotton-cuprammonium dispersions with fluidity values less than 10 rhes, that the velocity gradient adjustment is readily obtained in one discharge. 14 references.

Viscosity Measurement

The cupriethylenediamine viscosity method as a control in the manufacture of pulp of specified viscosity. Alfred A. Corey. *Paper Trade J.* **118**, 37 (May 18, 1944).

It was found that the cuprammonium viscosity test was not adequate for mill control so that the cupriethylenediamine method was adapted. This method has been used successfully; no details are given.

Relationship between cellulose viscosities measured by the TAPPI standard and the cupriethylenediamine methods. Alfred A. Corey. *Paper Trade J.* **118**, 51–2 (May 11, 1944).

Viscosities of a series of bleached sulphite pulps covering a range of high and low viscosities have been measured by the TAPPI standard and cupriethylenediamine methods. The results obtained by the two methods are compared on the basis of 1% cellulose solutions, and the relationship is shown graphically.

CHEMICAL AND PHYSICAL RESEARCH

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Alkyl Sulfates

The adsorption of sodium alkyl sulfates by wool and other fibers. R. G. Aicken. *J. Soc. Dyers and Colourists* **60**, 60–5 (Mar. 1944).

The adsorption of sodium alkyl sulfates by wool has been studied under a variety of conditions and it has been shown that it is the single long-chain alkyl sulfate ion which is involved. This ion reacts with the basic groups present in wool and behaves as a colourless dye, in that the adsorbed ions cannot be removed by a simple washing process. Quite large amounts of the sodium alkyl sulfates are adsorbed, viz. up to 2.5% on the weight of the fiber, under neutral conditions. The extent of the adsorption increases with increasing hydrogen ion concentration, and the concentration of the sodium alkyl sulfate solution is re-

duced to practically zero if there is less present initially than corresponds to the acid-combining capacity of the wool. The retarding and levelling action that these compounds exert in dyebaths can be explained in terms of this adsorption, since the dye sulfonic acids and the alkyl sulfates are competing for the same basic groups in the wool fiber, or active centers in other fibers.

The extent and the rate of adsorption is increased by the addition of electrolytes, the ion of sign opposite to that of the long-chain ion being responsible. As in the case of the effect on the interfacial tensions, the univalent ions fall into a lyotropic series. Measurements have also been made of the adsorption by other textile fibers, where similar effects have been observed. The extent of the adsorption has been shown to depend largely upon the number of basic groups present in the fiber.

Cellulose Solubility

The solubility of cellulose preparations in various solvents. *Jentgen's Kunstseide u. Zellwolle* **24**, 462–75 (Oct. 1942) (through *Bull. Inst. Paper Chem.*, 1944, **14**, 236–7).

Cotton, cotton linters and various rayon pulps were subjected to degradation with sulfuric and hydrochloric acids, with hydrogen peroxide and potassium bichromate-sulfuric acid, to aging (in the form of alkali cellulose) in the presence and absence of hydrogen peroxide, to acetolysis, and to disintegration in the oscillating mill. In addition, cellulose was regenerated without degradation from (1) amorphous cellulose triacetates (D.P. 18–200) which had been prepared with sulfuric-perchloric acid as catalyst and (2) fibrous triacetates (D.P. 700 and above) prepared by saponification with alcoholic postassium hydroxide in the presence of sodium sulfite. Both types of acetates were used in the form of fibers and precipitates. For comparison, the cellulose was regenerated from the fibrous triacetates directly, as well as from fibers and precipitates prepared from solutions and from acetate rayon. The degraded cellulose preparations and the regenerated celluloses were tested for solubility in aquous sodium hydroxide, calcium thiocyanate, sodium zincate, and formic acid-calcium chloride or formic acid-zinc chloride. The results obtained with the degraded cellulose preparations were compared at the same D.P. (200–300). It was found that those preparations which had retained their fibrous structure were almost completely soluble in calcium thiocyanate and sodium zincate, but only to a limited extent in formic acid-calcium of zinc chloride. The solubility in aqueous sodium hydroxide was more pronounced with the oxidized than with the hydrolized preparations. Disintegration in the oscillating mill facilitated solution and led to complete solution of cotton linters and refined rayon pulp in formic acid-calcium chloride after 8 hours, whereas with calcium thiocyanate and sodium hydroxide longer periods were required. The alkali solubility of celluloses regenerated from the nonfibrous triacetates decreased in direct relation with the D.P. The dried preparations were less soluble than when used in moist form. The same relationship between solubility and D.P. was shown by the celluloses regenerated from the fibrous triacetates, the solubility being lower for the films than for the fibers, and greatest for precipitates. The solubility of the latter approached that of celluloses regenerated from the non-fibrous triacetates. The D.P. of the non-fibrous acetates, as well as of the cellulose regenerated therefrom, may be determined by using aqueous sodium hydroxide in the place of cuprammonium hydroxide as a solvent. However, both the fibrous acetates and the regenerated fibers, even of relatively low D.P., were insoluble in this agent, whereas the fibrous acetates in the form of their precipitates were soluble up to a D.P. of 500. The solubility in formic acid-calcium (zinc) chloride showed similar trends. Cotton cellulose regenerated from its solution in phosphoric acid and having a D.P. of 1080 was completely soluble in formic acid-calcium chloride, but much less soluble in sodium zincate and calcium thiocyanate. The author interprets his results as showing that degradation alone does not suffice to impart to cellulose the peculiar solubility behavior characteristic for regenerated cellulose and that such behavior is brought about only by solution

and reprecipitation and, possibly, by far-reaching mechanical disintegration.

Color and Constitution

Color and constitution. Part VIII. The coupling of the four m-halogenophenols and the chromo-isomerism of the 3-halogeno-4-benzeneazophenols, explained on resonance theory. H. H. Hodgson. *J. Soc. Dyers & Colourists* **60**, 43–5 (Feb. 1944).

The unique mono-coupling of m-fluorophenol in 4-position, and the mono- and di-coupling of the other three m-halogenophenols in 4- and 2:4-positions, respectively, are discussed from the standpoint of hydrogen bonding and the theory of resonance. The consequent chromoisomerism which arises both in the 3-halogeno-4-benzeneazophenols and in 3-nitro-2-naphthylamine is adequately explained on the above theory.

Summary of available information of small color difference formulas. Dorothy Nickerson. *Am. Dyestuff Reptr.* **33**, 252–6 (June 3, 1944).

Six general formulas for specifying small color differences are examined, two based on the Munsell concept of hue, value, and chroma relations; two based on the Judd UCS diagram and a lightness scale that equals $\sqrt{Y}$; two based on Adams color space concept of "chromatic value" and "chromatic valence." Application of the first Munsell formula requires the least work, the Judd formula most. Results of formula differences as correlated against an average of visual observations rate the results by the Munsell formulas slightly higher than by other formulas. Accuracy of results by use of any of the formulas depends upon the relative accuracy of the original color measurements to which they are applied.

Why small color differences are important in textiles. F. T. Simon and E. I. Stearns. *Am. Dyestuff Reptr.* **33**, 231–5 (May 22, 1944).

This paper was the opening one of the 1944 spring Discussion Session of the Inter-Society Color Council on Small Color Differences and discusses the matter particularly in relation to the needs of the armed forces. The views presented by the authors on the need for improved color tolerances in the textile industry in regard both to color matching and the evaluation of fastness, were reviewed by members of the Color Committee of the American Association of Textile Chemists & Colorists and the paper may therefore be considered an authoritative presentation.

Fermentation Process

Aerobic decomposition of cellulose by thermophilic bacteria. H. C. Murray. *J. Bacteriology* **47**, 117–22 (Feb. 1944) (through *Bull. Inst. Paper Chem.*, 1944, **14**, 241).

It has been felt by previous investigators that thermophilic cellulose bacteria are, in general, anaerobic, because it is necessary to seal the plates used for agar preparations of these organisms and no growth occurs under normal aerobic conditions in agar. They imply that the cultivation in flasks is made possible by the action of associated organisms setting up anaerobic conditions. The present studies indicate that the difficulty with aerobic growth in agar of thermophilic cellulose-splitters is that of humidity. Saturation of the atmosphere seems necessary for optimum growth. Cultures of bacteria were readily obtained from soil and ruminant feces which attacked filter paper at 60° C. under aerobic conditions. These cultures were inhibited by several anaerobic methods. 11 references.

Interfacial Tension

The oil-water interface of mineral oil-polar compound mixtures. R. G. Aicken. *J. Soc. Dyers & Colourists* **60**, 41–3 (Feb. 1944).

The interfacial tension of various mixtures of mineral oil and polar compounds has been measured. From these results the packing of the polar compounds at the oil-water interface has been determined, and it has been shown that a stable 1:1 mineral oil-polar compound complex is present in the interface over a wide range of concen-

trations, regardless of the interfacial tension of the system. The presence of this 1 : 1 complex provides the basis of a simple mechanistic picture of the great stability of emulsions stabilised by mixtures of oil-soluble and water-soluble polar compounds, e.g., mixtures of higher alcohols and sodium alkyl sulfates.

The addition of glycerol mono-oleate reduces the interfacial tension of mineral oil against water to an extremely low figure, and it is effective in extremely low concentrations. Solutions of glycerol mono-oleate in mineral oil are shown to be micellar in nature; this is one of the few cases reported of the formation of micelles in hydro-carbon systems.

The interfacial tension of sodium secondary alkyl sulfate solutions against oils. R. G. Aicken. *J. Soc. Dyers & Colourists* **60**, 36–40 (Feb. 1944).

The interfacial tension of solutions of sodium secondary alkyl sulfates against a hydrocarbon fraction and other oils has been measured at 20 degrees C. There is a sharp break in the interfacial tension-concentration curve at the critical concentration, and the concentration at which this break occurs is quite independent of the oil phase. Good agreement was obtained between the values for the critical concentration obtained by this method and by a flotation method.

The addition of electrolytes to the sodium secondary alkyl sulfate solution has a very pronounced effect, reducing both the interfacial tension of the system and the critical concentration. This effect is due almost entirely to the ions of sign opposite to that of the long-chain ions, and in the system investigated it is a positive ion effect. Divalent ions have more effect than univalent ions, and the latter fall into a lyotropic series. The ions which approach closer to the interfacial film, i.e., the least hydrated ions, have the greater influence on the interfacial tension of the system.

The nature of the surface-active species has been critically discussed, and it has been shown that the facts can be satisfactorily explained on the basis that the single long-chain ion is the surface-active entity.

Protein Chemistry

The chemistry of protein denaturation. Hans Neurath, Jesse P. Greenstein, Frank W. Putnam, and John O. Erickson. *Chem. Rev.* **34**, 157–265 (Apr. 1944).

A comprehensive review of this subject is presented, the following phases of the problem being covered most thoroughly: the chemical reactivity of denatured proteins, the kinetics and thermodynamics of protein denaturation, the properties of denatured proteins, including molecular weight, molecular shape, hydration, electrochemical properties, structure, and immunological properties. Finally, the question of reversibility of protein denaturation is considered. 418 references.

Starch Acetate

Preparation and properties of starch acetate. Lee T. Smith and R. H. Treadway. *Chem. and Eng. News* **22**, 813–7 (May 1944).

Dry domestic potato starch was acetylated by (1) a mixture of acetic anhydride, acetic acid and a catalyst and (2) acetic anhydride alone after a preswelling with formic acid. Perchloric acid and sulfuric acid were used as the catalyst. Most experiments were run at 95° C for 9 hours. The solubility of starch acetate depends on the degree of substitution and extent of degradation. Degraded types with 5 to 15% acetyl content are dispensible in water. Pyridine and ethylene chlorohydrin are the best solvents for starch acetate of 30 to 40% acetyl. Method (1) requires a longer time and caused greater degradation than Method (2).

Moisture Absorption of Starch

Moisture absorptive power of starch hydrolyzates. J. E. Cleland and W. R. Fetzer. *Ind. Eng. Chem.* **36**, 552–5 (June 1944).

A method of obtaining absorption and desorption moisture equilibrium data for sugars and sirups has been developed. Starch hydrolyzates are effective materials for absorbing water. The amount of absorbed water increases with dextrose equivalent and with increasing relative humidity.

Starch hydrolyzates are compared with two common materials used as humectants, invert sirup and glycerol. The water content of each material, when at equilibrium at any relative humidity between 20 and 78%, is defined. When adequately dispersed, each material reaches a characteristic water content which is at equilibrium with an atmosphere of given humidity (or relative vapor pressure) at the same temperature. Hence, precise measurement of the vapor pressure should accurately define the water content.

Stress and Relaxation

Stress relaxation of natural and synthetic rubber stocks. A. V. Tobolsky, I. B. Prettyman, and J. H. Dillon. *J. of Applied Physics* **4**, 380–95 (Apr. 1944).

Measurements of stress decay as a function of time made at constant elongation on thin bands of gum and tread type natural rubber (Hevea), Neoprene, Butyl, Buna S. and Butaprene N stocks indicate that both secondary and primary bond relaxation occur. Practically complete relaxation is observed to take place in the experimental time (about 100 hours) at temperatures at and above 100 degrees C. The manner in which the rate of relaxation depends on temperature and the fact that the rate is independent of elongation and of the presence of carbon black in the vulcanizate indicate that stress decay is caused by a definite chemical reaction which deteriorates the rubber structure, and oxidative scission is suggested as the mechanism of deterioration of the primary bonds. The stress relaxation data, obtained over a temperature range from $-50°$ C to $+150°$ C, appear to verify modern concepts of the structure of elastomers. Theoretical equations are derived which give very good agreement with the observed relaxation data at high temperatures. The free energy of activation for the oxidative scission is found to be 30.37 kcal. per mole for the Hevea gum stock, and differs from this value by less than ± 2.0 kcal. per mole for all other stocks, indicating the same general reaction for all. However, these small differences in free energy of activation correspond to considerable differences in times of decay, which fact might be significant in evaluating the resistance of rubber stocks to deterioration.

Starch Studies

Water sorption by cornstarch. L. Sair and W. R. Fetzer. *Ind. Eng. Chem.* **36**, 316–9 (Apr. 1944).

Sorption isotherms of cornstarch and ten of its commercial modifications have been determined. Modification up to 60-fluidity thin-boiling starch results in apparently little hydration change. Dextrinization, with or without acid catalyst, results in products with different hydration characteristics. There is little correlation between the viscosity of the modified products and their hydration capacity. When compared to cornstarch, the modifications fall roughly into three groups with respect to sorption behavior: (1) those in which the ratio of water retained is independent of the humidity—e.g., British gum and chlorine-oxidized starch; (2) those in which the ratio varies with the humidity, the variation being at high and low humidities—e.g. 90-fluidity starch, white corn dextrin, canary dextrin, and hypochlorite-oxidized starch; and (3) those in which the ratio continually decreases with the decreasing humidity—e.g., Amijel.

Resonance

A theoretical discussion based on resonance theory of the function of the halogen in the 2-halogenobenzoquinones, the 3-halogenobenzoquinone 4-oxines, the melting point rule for nitroso-quinonoid isomerides, and the nitration of 3-fluoro-2: 4: 6-trichloro-anisole. H. H. Hodgson. *J. Soc. Dyers and Colourists* **60**, 65–7 (Mar. 1944).

The melting point order and variation of colour of the 2-halogenobenzoquinones are interpreted on resonance theory, which also satisfactorily accounts for melting point regularity among the 3-halogenobenzoquinone-4-oximes. Benzoquinonemonoximes and their ethers always melt at a higher temperature than the tautomeric nitrosophenols and ethers due to predominance of a more highly polarised resonance structure. The effect of fluorine in controlling

the nitration of 3-fluoroanisole and of its mononitro-derivatives may be readily explained by resonance structures, and the argument thus developed is applied satisfactorily to the order of replacement of all three chlorine atoms by nitro-groups when 3-fluoro-2:4:6-trichloroanisole is treated with fuming nitric acid at 0 degrees C.

Thermal Expansion

Thermal expansion and second-order transition effects in high polymers. Part I. Experimental results. R. F. Boyer and R. S. Spencer. *J. of Applied Phys.* **4**, 398–405 (Apr. 1944).

Experimental values of second-order transition temperatures T_m and of cubical expansion coefficients below and above this temperature are presented for several new materials, notably Saran. The thermal expansion behavior of two-component systems of incompatible materials (polystyrene plus polyolefins) has been studied. For relatively coarse dispersions (1000Å), T_m is 82° C, independent of composition, while the difference $\Delta\beta$ in cubical expansion coefficient above and below T_m is directly proportional to the volume fraction of polystyrene in the mixture. Unplasticized Saran behaves similarly in that T_m is constant while $\Delta\beta$ decreases linearly with increasing crystallinity. For molecular dispersions of incompatible materials both T_m and $\Delta\beta$ are functions of composition. It is shown that for most high polymers T_m increases with increasing intermolecular force constants, while the product of T_m and cubical coefficient of expansion above T_m is roughly constant (0.1 to 0.2).

Ultraviolet Light and Cellulose

The action of ultraviolet light upon cellulose. I. Irradiation effects. II. Post-irradiation effects. R. A. Stillings and R. J. Van Nostrand. *J. Am. Chem. Soc.* **66**, 753–60 (May 1944).

Cotton linters were exposed to the radiation from a General Electric Portable UVIARC outfit for 24 hours or more in atmospheres of nitrogen, oxygen, etc. The copper number, alpha cellulose, degree of polymerization and gas evolved were determined. Cotton irradiated in nitrogen showed considerable degradation. The rate of degradation increased with increasing oxygen content. The reaction does not appear to be a first order reaction. The changes initiated by irradiation in an atmosphere of nitrogen continue to occur during storage if air is present, but cease when air is absent. The post-irradiation effect was decreased if oxygen was present during irradiation.

Scale Substance of Wool

The scale substance of wool. Walton B. Geiger. TEXTILE RESEARCH **14**, 82–5 (Mar. 1944); J. of Research, Natl. Bur. Standards **32**, 127–30 (Mar. 1944);

The scaly outer layer of wool fibers is commonly believed to protect them from injury and to assist in the felting processes. Little is known about the composition of this layer because no ready means of separating it from the rest of the fiber has been available. Earlier work in this laboratory has shown that wool that has been reduced with thioglycolic acid and then alkylated with ethyl bromide is attacked by pepsin in such a way that the scale material remains intact while the interior of the fiber is completely dissolved. The composition of the scale material so obtained has now been studied. It has been found that it is essentially protein in nature and, although it contains the same amino acids as the whole wool, the proportions of these in the two materials differ.

BLEACHING: DYEING: FINISHING

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Dyeing of Nylon

Some theoretical and practical aspects of the dyeing of nylon yarns and fabrics. E. B. Abbot. *J. Soc. Dyers and Colourists* **60**, 55–9 (Mar. 1944).

A critical examination has been made of the dyeing properties of cellulose acetate dyes on nylon. Times of half-dyeing for 20 dispersed dyes on nylon and on acetate indicate that rates of dyeing are approximately the same on the two fibers. Percentage exhaustion of dispersed dye on

nylon decreases as initial concentration of dye is increased. Degree of exhaustion was not greatly affected by variation in the liquor-goods ratio between 25:1 and 100:1. The temperature range (considered good if degree of exhaustion varies little between 55° C and 85° C, and poor if exhaustion increases steadily with temperature) was found to agree well with rate of dyeing properties, the rapid dyeing dyes having good, and the slow dyeing dyes having poor, temperature ranges. Temperature range information known for cellulose acetate holds equally well for nylon. The following points of comparison between cellulose acetate and nylon are noted: (1) in pale to medium shades dispersed dyes dye nylon and acetate at about the same rate but heavy shades do not build up as well on nylon as on acetate, (2) in all cases solubility of the dispersed dyes in cellulose acetate was greater than in nylon, roughly about twice as great, (3) the resultant color of a dye on nylon and on acetate may be very different, oranges being redder and reds being bluer on nylon, (4) dyes on nylon are generally slightly less bright on acetate, (5) usually fastness to light is somewhat less on nylon, (6) washing fastness at 40° C is about equal on the two fibers, (7) tendency to redistribute or level is much greater on nylon than on acetate, rapid dyeing dyes being most marked in this respect, (8) sublimation of dispersed dyes is at least as great on nylon as on acetate, (9) dyes subject to gas fading on acetate are but very slightly affected on nylon. Water soluble acetate dyes behave quite differently toward nylon and acetate particularly in that they show no salt sensitivity applied to nylon and that they have much greater affinity for nylon. Water soluble acetate dyes do not sublime from nylon material.

Wool Dyeing

Dyes for woolen hosiery yarns and fabrics, with some notes on the effect of chlorination. H. Crook. *J. Soc. Dyers and Colourists* **60**, 33–6 (Feb. 1944).

The scarcity of chrome has brought about development of wool dyeing formulas economical of this material; the chrome mordant method, comparatively wasteful of chrome, is used only when necessary for essential goods. Of other metals investigated as possible substitutes for chrome only copper shows any promise and it only in combination with chrome. The chromate process in hosiery dyeing is growing in importance due to its efficiency, and manufacturers are striving to increase the number of their chromate-dyeing dyes. The chromate-dyeing efficiency of a chrome dye may be judged by its ability to dye wool from a neutral dyebath. On cellulose acetate-rayon-wool unions very good all-round fastness is obtained by dyeing with combinations of dispersed acetate dyes and Carbolan dyes. It is suggested that the dullness and unevelness often occurring in dyeing with the sulphocyanine type of dyestuff may be caused by residual alkali in the material after scouring and by too long immersion in the boiling dyebath. The great bulk of service hose is now given some type of non-shrink finish and this results in a marked change in the reaction of the wool toward dyestuffs, in most cases the affinity being markedly increased. For example non-shrink wool may be dyed by the chromate process by chrome dyes showing no chromate-dyeing properties on ordinary wool. Wools damaged in scouring, in the non-shrink treatment, or by other means, and which would normally be practically impossible to dye level may be dyed evenly if previously treated with hydrogen peroxide, for example, two-volume hydrogen peroxide buffered to pH 9.5–10, for three hours at 40° C. This treatment is also very valuable in dyeing tippy wool or wool from different sources of widely varying affinity.

Fabric Coatings

Lacquer-type fabric coatings. Dorman Mc Burney. *Modern Plastics* **21**, 93–5, 172–4 (Jan. 1944).

The type of coatings used and the requirements of the following military fabrics are given: Army raincoat material, outer shell for sheepskin jackets, clothing interliner, utility cloth, mountain tent material, adhesive tape backing, hospital sheeting, jungle hammock canopy, blanket covering, Navy bedding bag, life preserver cover, dye pouch material, upholstery material, kit

covers, and aeronautical products. 18 references.

Flameproofing Textiles

Modern flameproofing for textiles. Anon. *Silk and Rayon* **18**, 445 (Apr. 1944).

The characteristics and advantages of sodium borophosphate as a flameproofing agent are: non-crystalline film that does not dust off, is not removed by drycleaning, does not affect color or luster of fabric, does not change the hand of the fabric, is non-poisonous, does not give off fumes or odors when heated, is soluble in water, and is free from strong acids or alkalis. The proper concentrations and methods of application for a variety of fabrics are given.

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Improvement of Cotton

Farm and gin preparation practices affecting cotton spinning value. Francis L. Gerdes. *U. S. Dept. of Agriculture, Bulletin* (Apr. 19, 1944).

The need for preserving and improving the spinning quality of cotton is gaining rapid recognition in face of labor shortages on farms and in gins. In an address prepared by Francis L. Gerdes, In Charge, Stoneville Laboratory, Cotton and Fiber Branch, War Food Administration, for delivery at the Cotton Textile Conference, Nashville, Tenn., April 19, farm and gin preparation methods that affect spinning quality were discussed. Fiber properties that are important from a spinner's standpoint were enumerated. It was pointed out that, while it has already been demonstrated that many fiber properties are inherent characteristics of the leading varieties of cotton, the spinning results with a particular variety may be modified to a considerable extent by farm and gin practices. Gin practices that affect fiber length distribution, foreign matter content and preparation of the lint were discussed. The methods which growers employ in harvesting and handling cotton have a direct bearing on cotton spinning quality and manufacturing performance. Excess foreign matter and moisture in the seed cotton influence the grade of the lint which, in turn, affect its spinning value. The extent of weather damage to the unpicked cotton in the field is directly related to the more important spinning quality elements of the ginned lint such as manufacturing waste and yarn strength and appearance. Loss in spinning value associated with weather damage is seldom recoverable. Nevertheless, mechanical driers and cleaners, when properly operated in gins, have been found to bring about improvement in the grade of cotton requiring such gin treatment with a consequent lowering of manufacturing waste and preservation of yarn quality. Agricultural leaders are now urging prompt picking to preserve inherent fiber qualities, recognizing the fact that an added burden is being placed on the ginner. Timely picking of cotton was reported to improve the cotton fiber and spinning quality as compared with picking after undue field exposure. Preliminary data given in the address in connection with the discussion of mechanical cotton picker developments indicate that the picker has promise of being successful. It was reported that present-day cleaning and extracting equipment is not very effective in cleaning mechanically-picked cotton which contains green- or dry-leaf particles and sticks and stems. In ginning tests, the machine-picked cotton subjected to extensive gin cleaning provided lint which averaged almost 2 grades lower in quality and 4 percent higher in foreign matter content than hand-picked cotton. However, in spinning tests, the machine-picked cotton yielded enough of the foreign matter in the form of picker and card waste to cause the yarns spun from the cotton to be equally as good in quality as that of the yarns spun from the hand-picked cotton.

Wool Shrinkage

1943 wool shrinkage studies in the Department of Agriculture. Alexander Johnston, Warner M. Buck, and George C. Le Compte. *National Woolgrower* (Apr. 1944).

During 1943 core samples were taken from 100 individual clips of domestic wools and from a few graded lines, using a new coring device which is described. These clips to-

taled more than three million pounds and came from 13 major wool-growing states of the West. An average of 10 test bags were taken at spaced intervals from each of these clips. In the majority of cases 10 cores were taken at random from—or at spaced intervals from—each bag. These core samples were scoured by commercial scourers. On 92 of the one hundred lots tested, estimated shrinkages had been assessed by committees each consisting of three competent wool dealers commissioned by the Government for that purpose. It is shown that for the 92 lots on which both estimates and core samples were obtained: (1) for the core samples, the range of errors extends from a low of —4.4 percent to a high of 3.8 percent with an average error of 1.166 percent. For the estimates, the range of errors extends from a low of —10.7 percent to a high of 9.4 percent with an average error of 2.82 percent. From the above and from detailed analysis of the test results, it is apparent that the core shrinkages more accurately expressed the true or actual shrinkages than did the estimates.

Calculating Roving Production

Speed and accuracy in calculating roving production. Fritz Zweifel. *Textile World* **94**, 71–8 (May 1944).

Production tables for roving frames now generally used make a standard allowance of 15 minutes per set for doffing and other incidental shut-downs. Tables thus computed are not, in the author's opinion, correct. Time allowed for doffing and incidental shut-downs should be gaged accurately if the production is to be forecast with any degree of accuracy. Incidental stops include: time required for doffing; interference, or time lost on a machine because of inability of the operator to service it immediately; time lost on a machine because of inability of the operator to service it immediately; time lost in piecing broken ends; week end cleaning, and time lost in picking flyers in cases where excessive shedding requires doffing flyers between regular doffs. Production tables are given. The cotton tables were figured on the basis of a 1⅛ inch front-roll and 1.30 twist multiple, and the rayon tables with 1⅜ inch front-roll and 1.00 twist multiple. Spindle speeds for both cotton and rayon are: 850, 950, 1050, and 1100 r.p.m. for 10×5, $9 \times 4\frac{1}{2}$, 8×4, and $8 \times 3\frac{5}{8}$ slubbers, respectively. By following a few simple rules and using conversion formulas (which are given) these tables can be converted to fit every existing condition.

Applications for Twist Multiple Formula

Extension of the conventional twist multiple formula to new materials. S. L. Gerhard. *J. Applied Phys.* **15**, 474–6 (June 1944).

Mathematical relations between a few geometric yarn properties are used to develop the concept of relative density. With the aid of this idea it is possible to deal with yarns made of different materials but having the same relative density as though they were made of the same material, so far as their geometric properties are concerned. A simple formula is derived to convert twist multiples from one yarn count system to another. The figure obtained for the twist to be put into the new yarn is only an "educated guess," but it is hoped that it will shorten the amount of experimentation necessary for determining the optimum twist in new yarns.

Milling Temperatures

On the existence of a critical temperature for milling. J. B. Speakman, J. Menkart, and W. T. Liu. *J. Text. Inst.* **35**, T41–52 (Apr. 1944).

Using solutions of soap, borax and sulfuric acid as milling agents, the rate of shrinkage of an all-wool woven fabric in a model milling machine has been determined at different temperatures up to 60° C. The optimum temperature for milling with solutions of soap and borax was found to be 35° to 37° C, but no evidence could be obtained for the existence of a critical temperature for acid milling. These results lend further support to an earlier theory of milling, for whereas the hysteresis between extension and contraction of wool fibers in alkaline media increases, the hysteresis in acid media decreases steadily with rise of temperature above 25° C.

Water-Vapor Permeability

Water-vapor permeability of certain coated fabrics. John T. Stearn and Albert S. Cooper, Jr. *Am. Dyestuff Reptr.* **33**, 150–56 (Mar. 27, 1944).

A new and practical concept is presented of the cooling effect afforded by waterproof garments when certain types of coatings are employed. Coating materials to be suitable for this purpose should have a relatively high permeability for water vapor while preventing the passage of liquid water. The results presented indicate that several synthetic resins and cellulose derivatives should be satisfactory if they are not specifically compounded to make them too highly water resistant. As an aid in such work the well known permeability cup test has been simplified to a method which does not require highly specialized or unavailable equipment, or an undue amount of the operator's time. It is shown to be amply accurate for the present purpose but subject to further refinement for testing the permeabilities of packing materials, and other moisture resistant coatings that are especially compounded for low water permeability. By means of this test it is shown that the permeability to water vapor of uncoated cotton broadcloth, for instance, is reduced surprisingly little by application of a coating which withstands the 50-cm. hydrostatic pressure A.A.T.C.C.

MISCELLANEOUS

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Phosphorescence

The phosphorescence of textile fibers and other substances. (Continued from Nov. 1943.) H. E. Millson. *Textile Colorist* **66**, 109–11, 132 (Mar. 1944).

The fluorescent and phosphorescent properties, which may in some cases be used as qualitative tests for purity, of a considerable number of chemical compounds are listed. Phosphorescence may also be useful in fiber identification, detecting and identifying chemical damage, and in checking process efficiency. For example, in Aralac and rayon union fabrics where complete desizing is essential, incomplete removal of size may be detected readily from measurement of duration of phosphorescence. Phosphorgraphs may be made by exposing a photographic plate to the phosphorescence emitted by an object which has been exposed to ultraviolet while partially covered by a non-phosphorescing (metallic) object, thus producing an outline of the latter on the plate. A number of phosphorgraphs are illustrated.

Neoprene

Neoprene and other synthetic rubbers in the textile industry. Emil H. Krismann. *Am. Dyestuff Reptr.* **33**, 246–51 (June 5, 1944).

A brief history of the development of synthetic rubbers and description of their properties and uses. Paper presented at a meeting of Northern New England Section, A.A.T.C.C. The discussion reported brought out pertinent information.

Peanut Oil

The preparation and properties of sulfonated peanut oil. John B. Gallent. *Am. Dyestuff Reptr.* **33**, 148–9, 156 (Mar. 27, 1944).

Some experiments in which refined peanut oil was sulfonated by several methods are discussed. A mixture of sulfuric and fuming sulfuric acids gave the best sulfonation in the laboratory though of doubtful use in industry. The sulfonation of "fractionated" peanut oil gives promise provided such "tailored" oils can be economically produced in quantity. To improve the appearance and reduce possibility of rancidity, a blend of 75 percent peanut oil and 25 percent castor oil was sulfonated. The resulting product was clear and water soluble—although saturated fatty acids did separate on standing. In general, sulfonated peanut oil is easily produced, is a good emulsifier, and when not too old and when treated with an inhibitor (such as soybean lecithin) does not develop odor when applied to textiles, and gives otherwise satisfactory results.